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SVHSER17066
REV. A

FINAL TEST REPORT

FOR

URINE PRETREAT INJECTION SYSTEM

AUG 1995

PREPARED UNDER CONTRACT NO. NAS 8-38250-28

BY

HAMILTON STANDARD SPACE SYSTEMS INTERNATIONAL

DIVISION OF UNITED TECHNOLOGIES

WINDSOR LOCKS, CT 06096

FOR

NASA MARSHALL SPACE FLIGHT CENTER

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DOCUMENT REVISION HISTORY

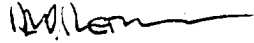
Rev. Ltr.	Page(s) Affected	Description of Revision	Approval Signature and Date
A	17	Change "2:1" to "1.8 :1"	 8 SEPT 95
	33	In Table A change "6.8" to 0.8" (2 places)	

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SUMMARY

A new method of introducing the OXONE® Monopersulfate Compound for urine pretreat into a two-phase urine/air flow stream has been successfully tested and evaluated. The feasibility of this innovative method has been established for purposes of providing a simple, convenient, and safe method of handling a chemical pretreat required for urine processing in a microgravity space environment. Also, the Oxone portion of the urine pretreat has demonstrated the following advantages during real time collection of 750 pounds of urine in a Space Station design two-phase urine Fan/Separator:

- Eliminated urine precipitate buildup on internal hardware and plumbing.
- Minimized odor from collected urine.
- Virtually eliminated airborne bacteria.

The urine pretreat, as presently defined for the Space Station program for proper downstream processing of urine, is a two-part chemical treatment of 5.0 grams of Oxone and 2.3 ml of H_2SO_4 per liter of urine. This study program and test demonstrated only the addition of the proper ratio of Oxone into the urine collection system upstream of the Fan/Separator.

This program was divided in three major tasks: (1) A trade study, to define and recommend the type of Oxone injection method to pursue further, (2) The design and fabrication of the selected method, and (3) A test program using high fidelity hardware and fresh urine to demonstrate the method feasibility.

The trade study was conducted which included defining several methods for injecting Oxone in different forms into a urine system. Oxone was considered in a liquid, solid, paste and powdered form. The trade study and the resulting recommendation were presented at a trade study review held at Hamilton Standard on 24-25 October 94. Representatives from NASA/MSFC, ION, Boeing, and Hamilton Standard/Huntsville were in attendance. An agreement was reached at the meeting to continue the solid tablet in a bag concept which included a series of tablets suspended in the urine/air flow stream. These Oxone tablets would slowly dissolve at a controlled rate providing the proper concentration in the collected urine.

To implement the solid tablet in a bag approach, a design concept was completed with prototype drawings of the complete urine pretreat prefilter assembly. A successful fabrication technique was developed for retaining the Oxone tablets in a fabric casing attached to the end of the existing Space Station Waste Collection System urine prefilter assembly. The final pretreat prefilter configuration held sufficient Oxone in a

tablet form to allow normal scheduled daily (or twice daily) change out of the urine filter depending on the use rate of the Space Station urine collection system.

The actual tests to prove the concept were conducted using the Urine Fan/Separator assembly that was originally used in the STS-52 Design Test Objective (DTO) urinal assembly. The Fan/Separator was installed into a portable urine collection test rig assembly that was positioned in a men's bathroom at Hamilton Standard for real time collection of urine. Urine collection continued over a 2-month period with a total of 40 batches of urine collected. Each batch consisted of approximately one day of urine collection with a fresh pretreat prefilter installed at the beginning of the day. Based on a mass balance of urine collected and Oxone tablets used, an average pretreatment ratio of 5.2 grams of Oxone per liter was obtained by the controlled dissolution rate method of dispensing pretreat. This meets the test program goal of a minimum average of 5.0 g per liter. Also, a post test teardown of the test hardware including the urine separator and downstream urine check valve showed that Oxone alone does an excellent job of eliminating undesirable urine participates in the system. The average pH maintained in the collection reservoir was about 4 1/2.

Other related tests were conducted to demonstrate the actual minimum ratio of Oxone to urine that will control microbial growth. The results of this test indicated that at 3.0 gm/l there was limited control and above 4.0 gm/l microbial growth was prevented over 7 days. Also, a microbial air sampling test of the Urinal Fan/Separator 10 cfm air entrainment flow indicated that sample points within the urine collection system and at the outlet did not contain microbes over the detection limits of 12 CFU per M³. Air samples at the urine funnel showed that men's bathroom air entering the system was as high as 193 CFU per M³.

INTRODUCTION

There are several problems and considerations for the proper collection, storage and processing of urine in a microgravity environment for long duration manned space craft missions such as the Space Station. Urine processing for water reclamation usually requires the addition of chemical to fix the urea, provide microbial control, and minimize urine particulate deposits. Also, since there is minimal use of flush water, the additive chemicals are required to eliminate particulate deposits in equipment and plumbing which can cause premature failure of hardware and systems. Since the original conception of Space Station system layout the urine pretreat chemical additive has been defined as oxidizer of Potassium Monopersulfate Compound (Oxone) and a concentrated solution of Sulfuric Acid (H_2SO_4). The present requirements for concentration ratios with urine is 5.0 grams of Oxone per liter of urine and 2.3 ml of H_2SO_4 per liter of urine. These ratios are driven by the downstream urine processing system to fix the urea and eliminate urine precipitates. These pretreat chemicals have been considered to be introduced at various points in the urine collection and processing system. Considerations such as crew safety, ease of handling, envelope volume, interface, and maintenance all enter into the decisions. The present system approach is to introduce the Oxone as a mixed solution of H_2O and Oxone powder upstream of the urine separator and inject a concentrated solution of H_2SO_4 directly into the urine outlet line downstream of the urine separator. Part of the decision to introduce only the Oxone upstream of the separator was to minimize the potential hazard of H_2SO_4 injection in the proximity of urine collection from the body. Also, it was established that injection of Oxone alone upstream of the separator was sufficient pretreat to keep the urine collection, separator and associated hardware and plumbing clean. It is obvious from the results of Shuttle STS-65 post flight urine separator hardware examination that collection of urine with no pretreat protection can cause serious quantities of urine deposits to accumulate on various surfaces. Figure 1 shows the Shuttle EDO WCS urine separator inlet housing after a 14-day STS-65 mission with only about 100 Kg (220 lbs) of urine collected. Figure 2 shows the end of the 0.118 inch internal diameter pitot tube almost completely plugged with foreign debris and urine deposits.

Previous testing at Hamilton Standard with liquid Oxone injection systems have already demonstrated that Oxone alone injected upstream of a urine separator will provide sufficient pretreatment to keep the separator, plumbing and downstream hardware (i.e., check valves, disconnects and plumbing) clean. A 90-day urine collection test was completed on 27 June 89 in which a total of 2976 lbs of urine was successfully processed through an Representative Environmental Control System (RSECS) Urine Separator with upstream liquid Oxone injection. Post test teardown demonstrated that the internal hardware, which was primarily stainless steel, was kept free of urine deposits with only the Oxone pretreat.



Figure 1 Shuttle STS-65 Post Flight Inspection of Urine Separator
Inside Surfaces Showing Excessive Urine Deposits After
Approximately 100 Kg (220 lbs) of Urine Without Pretreat

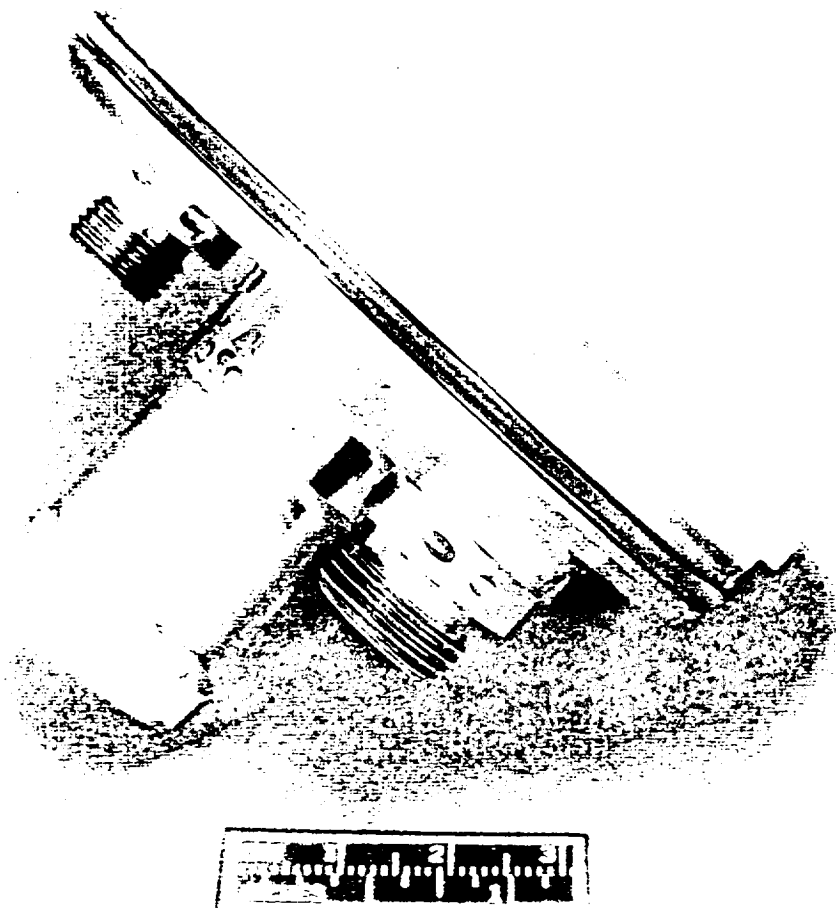


Figure 2 Shuttle STS-65 Post Flight Inspection of Urine Separator Pitot Tube Showing the Pitot Inlet 90% Plugged with Urine Deposits & Foreign Debris

The addition of Oxone to urine as the first step in the urine pretreat process does provide partial pretreatment with the following results. As an "oxidant", anti-microbial control is achieved. The urine solution pH is lowered to about 4-5 which does fix urea for room temperature conditions. The second step for full pretreatment of urine for the downstream urine processing at elevated temperatures is the addition of H_2SO_4 which lowers the pH to approximately 2. For information the DuPont Product Specification and Data Sheet for Oxone is included in Appendix C. The Material Safety Data Sheet (MSDS) for Oxone is included in Appendix D.

DISCUSSION

The combined effort for the first phase of the urine pretreat injection system investigation and test program was conducted for NASA/MSFC and ION. This first-phase effort specifically addressed adding the Oxone® portion only of the urine pretreatment chemicals and included the following major tasks:

- Trade study for various Oxone injection concepts.
- Design and fabrication of prototype hardware of the selected concept.
- Prototype test verification.

Trade Study - The urine pretreatment trade study began on 1 September 94 with the "brainstorming" of various methods of introducing Oxone into a two-phase urine/air flow stream in a microgravity environment. The trade study included dispensing of the Oxone in different forms such as liquid, powder, paste and solid shapes. The trade study included references and data from the early phase of the Commode/Urinal System (C/US) development effort before the C/US portion of the task was put on hold. At that point the baseline C/US schematic (SVSK117924) had defined the Oxone to be injected as a liquid upstream of the urine separator. Refer to the schematic in Figure 3. Some effort had been started to look at other injection concepts and these were incorporated into the trade study.

The first trade study compared seventeen Oxone pretreatment concepts which were composed of variations of Oxone-solution based systems, solid Oxone based systems, systems which used powdered Oxone, and a system which used an Oxone paste. The basis of the comparisons was a list of twenty "design & rating criteria." After review of this initial trade study, NASA & HSSSI chose seven of the original seventeen concepts for further, more detailed evaluation. This resulted in the second trade study. This trade study suggested that a filter integrated solid Oxone-based system was the most viable given the rating criteria, which was the same used for the first trade study. Of the seven concepts chosen for further evaluation, none was a liquid (Oxone solution) injection-type system. However, during a subsequent phone conversation between NASA, Boeing, and HSSSI, it was agreed that an automatic Oxone-solution injection

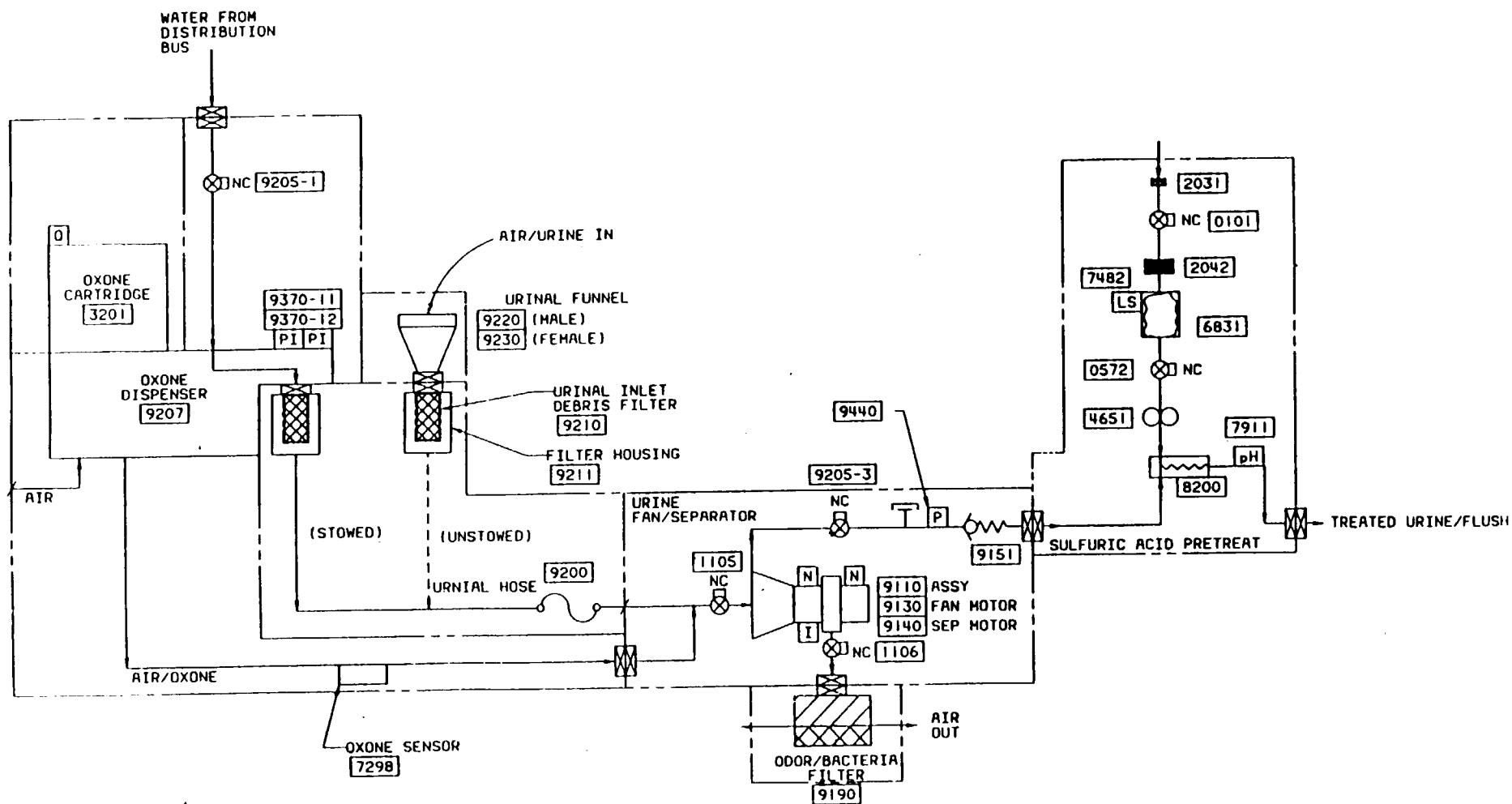


Figure 3 Space Station Schematic (SVSK117924) Showing Last Baseline Version of the C/US with the Liquid Oxone Injection Method.

system should be evaluated more carefully as compared to the solid Oxone system. This was, in part, because an automatic Oxone-solution injection system had already been presented at an earlier PDR as the baseline system. This resulted in the third "trade study" being performed.

A review of the three trade studies shows that a passive method of urine pretreatment which uses a 24-hour supply of solid Oxone integrated into the urinal filter is superior in almost all of the twenty-one rating criteria to powder, paste, and solution systems. The benefits of this type of system, hereafter referred to as the "tablets in-filter based system," is given below with respect to each of the rating criteria.

- **Simplicity of Design** - The tablets in-filter based system is by far the simplest design with the exception of a manual Oxone pill method, which would require the astronaut to insert an Oxone pill into the urinal before each micturation. Given a C/US without any pretreatment system, only the filter bag need be modified to accommodate the solid Oxone for this system to be incorporated. This would involve lengthening the bag about 3-4 inches to accommodate the 24-hour supply of solid Oxone rods ($\sim 1.1 \text{ in}^3$) and then cinching or seaming the bag above the Oxone to insure it remains in the bag. (Note: a solid Oxone tablet is actually a combination of powdered Oxone and a binder, probably polyethylene glycol, pressed into a cylindrical form.) Many of the other systems evaluated required pumps, tanks, sensors, injectors, electronics, and other mechanisms, in addition to modifications to the existing urinal housing.
- **Simplicity of Use** - The tablets in-filter system is the simplest of all the systems to use because it doesn't require the astronaut to do anything new. The Oxone supply is automatically replenished every 24 hours because it is *already* a requirement that the astronaut replace the filter every 24 hours.
- **Manufacturing Costs** - Very low. Cost additions to a C/US with no pretreatment system would include the manufacture of the solid Oxone rods and their integration into the filters, both of which are expected to be fairly simple.
- **Handling Ease** - The tablets in-filter based system will provide excellent handling ease. There will be no bulky injectors or other mechanisms attached to the side of the urinal housing to get in the way of the astronaut. Placement of the Oxone loaded filter into the urinal assembly should be no problem as long as the fan is ON, drawing air through the urinal hose.
- **Safety** - The safety of the tablets in-filter based system should be as good or better than the other systems. According to the DuPont Technical Information brochure, OXONE Monopersulfate has a low order to toxicity when taken internally. The lethal dose for rats is 2250 mg/kg. Oxone is irritating to the eyes, skin nose, and throat due to its acidity and oxidizing properties. DuPont observes an airborne exposure limit to Oxone dust of 1 mg/m^3 , 8-hour time weighted average. There should be far

less opportunity for the environment to become contaminated with a solid system than with a liquid or powder-based system, due to the inherent self-containment of the solid system. The polyethylene glycol binder used in creating the Oxone solid forms a wax-like material which precludes the likelihood of any Oxone dust forming and getting into the air. In addition, the Oxone solid is contained within the tight mesh of the filter, through which any Oxone particles are unlikely to pass. Two-fault tolerant containment will be provided for both new and used filters. As is currently done when handling a used urine filter on the Shuttle (*for hygiene purposes*), it would be advisable to use rubber gloves when removing the used filters, as some undissolved Oxone may remain in the filter.

- **Reliability** - With no mechanisms, sensors, or electronics to fail, the tablets in-filter based system should be the most reliable.
- **Ease of Maintenance** - The tablets in-filter based system is essentially a disposable system, therefore, it requires no maintenance.
- **Maintenance time** - None.
- **Envelope of System** - The tablets in-filter based system adds no envelope to a C/US without a pretreatment system.
- **Weight of System** - The tablets in-filter based system adds no weight to a C/US without a pretreatment system.
- **Envelope of Logistics Supplies** - The envelope of the filters would probably double due to their increased length and Oxone load. The new and used filter containers may also increase in size, depending upon the requirements of two-fault tolerant containment. Still, the tablets in-filter based system is expected to have the smallest logistics supply envelope of all the systems.¹ Additional weight will include the weight of the solid Oxone itself (about 47 gms/day), a tiny amount of weight from increased filtration media, and any additional weight caused by the need for two-fault tolerant containment for the new and used filters.
- **Ease of Retrofit into Orbiter (Adaptability for Use with Citric Acid)** - The use of citric acid as a pretreatment is the current baseline for the Shuttle. The tablet in-filter based system should be as adaptable for use with citric acid as any of the systems evaluated. Citric acid is easily formed into a dissolvable solid and is expected to require a smaller volume per dose than Oxone. (See the "Air Flow Impedance" criterion.) Also, this system requires no additions or modification to the existing Orbiter WCS and EDO WCS hardware, except for the modest filter redesign. (Note: EDO = Extended Duration Orbiter)

¹ Except for the manual Oxone pill system.

- **Usability in ISSA Commode/Urinal** - Expected to be the best system, due to its simplicity.
- **Material Compatibility** - None of the Oxone systems are expected to pose a material incompatibility problem with the rest of the C/US. However, unlike some of the other systems evaluated, the tablets in-filter based system cannot pose any materials incompatibility problems to itself simply because it adds no new hardware or materials to the C/US.
- **Development Risk** - The tablets in-filter based system has the lowest development risk of all the other systems.¹ The formation of the Oxone into a solid form has already been demonstrated. The formulation and geometry of a solid containing 42 grams of Oxone must be developed such that it dissolves fairly uniformly over a 24-hour period of average, intermittent micturations.
- **Development Cost** - Similarly, the tablets in-filter based system should have the lowest development cost associated with it.¹
- **Air Flow Impedance** - Although for this category the tablets in-filter based system ranks substantially higher than most of the powder or granular based "tea-bag" systems, this is the one category where it ranks lower than the paste or liquid systems. However, both analysis and actual testing has shown that the additional pressure drop caused by a rod loaded filter compared to an unloaded filter is only about .25 in. H₂O, or less than a 1 CFM drop in flow given an unloaded flow rate of 10 CFM.
- **Power Consumption** - The tablets in-filter based system uses no power.

The trade study was completed and presented at a formal review on 24-25 October 1994. The selected concept for further investigation and test was the solid Oxone/binder tablets held in a permeable membrane casing. A copy of the trade study as presented is included in Appendix A.

The most significant improvement in the Oxone tablet during the trade study was the identification of using Polyethylene Glycol (PEG) as the binder over the previously selected (plasdone) binder. Based upon some preliminary benchtop tests during the trade study PEG appeared to demonstrate better controllability over the dissolution rate with a liquid flowing over the tablet. Moreover as the tablet dissolved it reduced in size more uniformly from the outer surface and did not erode into pieces like the Oxone with the plasdone binder. This feature along with the other discriminators of the trade study provided the backup necessary for the decision to use the solid Oxone tablet with the PEG binder.

¹ Except for the manual Oxone pill system.

Design and Fabrication - The design phase of the program started with the concept sketches of the selected approach and expanded the drawing definition to the detail necessary to fabricate the required hardware for the final test phase. In the design phase several areas of concentrated effort were completed. These included the following: (1) Definition of new configuration prefilter housing, (2) Layout of the pretreat prefilter assembly to contain the tablets, and (3) tablet dissolution rate. The following is a list of design requirements and assumptions used for the design and test of the selected concept for the Oxone pretreat method:

- ◆ Pretreat system must introduce Oxone upstream of the separator.
- ◆ System will be used in conjunction with a sulfuric acid injection system downstream of the urine separator.
- ◆ 1.5 grams of Oxone required per average (300 ml) micturation.
- ◆ Space Station Urine Process Rate is 3.44 lbs urine/day/crew member. (1.56 liters/day).
- ◆ Flush Water is 1.09 lb/day/crew member (test at 80 ml/flush).
- ◆ Twenty-eight micturations per 24 hour period for a four member crew.
- ◆ 270 day total logistics storage timeline.

Urine Prefilter Housing Design - The present design of the urine prefilter housing for the EDO WCS is basically the same as the non-EDO version of the WCS. It has a nominal 7/8" internal diameter to match the urine collection hose and is hinged to allow insertion of a urine prefilter that is changed daily or twice a day depending on the use rate. (Reference Figure 4). The new solid Oxone tablets inserted into the hose causes an increased airflow restriction and a higher flow ΔP . A corresponding loss in air entrainment airflow would reduce the effective fluid (urine) containment. To offset the effect of reduced airflow area in the hose, it was decided to enlarge the I.D. of the existing housing to 1 inch. The increase in I.D. necessitated an inlet housing redesign which, simplified the original multi-piece hinged configuration (reference Figures 5 & 6) to a single detail part. The features of this redesign are several:

- 1) The hinge joint with the finger latch for insertion of the prefilter seal is eliminated.
- 2) The prefilter seal now fits into the funnel end of the housing and doubles as a non-metallic seal between the housing and funnel.



Figure 4 Existing Shuttle WCS Configuration of the Various Hardware Associated with Urine Collection at the Funnel and Prefilter Area



Figure 5

Comparison Between the Existing Hinge-Type Inlet
Prefilter Housing and the Single Piece UPIS Redesign

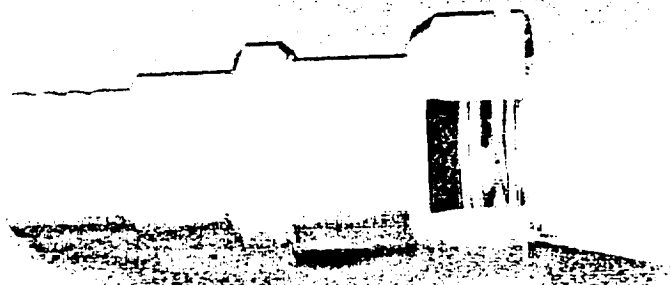
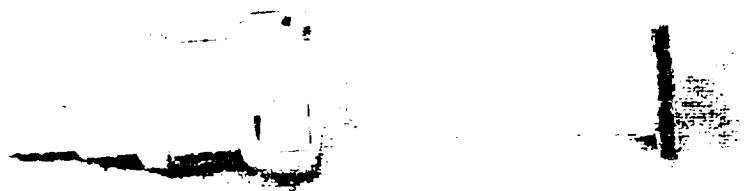


Figure 6

Exploded and Assembly Views of the Prefilter and Funnel
Along with the Redesigned Prefilter Housing (Reworked
Old Configuration C/US Prefilter Used for Demonstration)

- 3) The original metallic seat with the backup wave spring has been eliminated which minimizes trapped residual urine and its associated cleaning problems.
- 4) The O.D. of the prefilter housing remained at the same size such that the mating yoke that holds the housing would not be affected.
- 5) Finally, with the large housing I.D. a prefilter with a greater surface area and a corresponding lower ΔP can be utilized.

A detail drawing of the new prefilter housing (SVSK116871) is shown in Appendix B. In addition to a simpler design and the stated functional advantages, the new prefilter housing only weighs 127 grams (0.057 lbs) which is over a 40% weight savings over the old design housing of 215 grams (0.097 lbs.).

Urine Pretreat Prefilter - The design effort also included drawing definition of the prefilter/Oxone assembly. A cross section view is shown in Figure 7 and the complete drawing is enclosed in Appendix B. Once the trade study decision was made to use the solid tablet in a casing concept it was a matter of design definition to consider the various requirements for proper operation, ease of manufacturing, selection of materials and sizing. The size of the tablets; ie., length and diameter, was based on several issues. The tablet assembly had to fit into an existing 7/8" I.D. urine hose without significantly decreasing the flow cross sectional area and restricting airflow. The existing laboratory pill press was 0.5 inches in diameter. A wood mockup dummy was fabricated of the prefilter/Oxone assembly using 1/2" dowels as tablets. The mockup was hung in the benchtop airflow rig with a 7/8" I.D. Lexon tube with 10 CFM of airflow which demonstrated that the slight increase in ΔP was tolerable. This test was conducted prior to availability of the new designed larger 1.00" I.D. prefilter housing which slightly decrease the ΔP . Recognizing that the tablets would increase the ΔP , the goal was to offset this effect with the slightly larger I.D. urine inlet housing. This allows a larger urine inlet prefilter with more filter surface area and less ΔP .

Once the diameter of the Oxone tablet was established, the overall length of the prefilter/Oxone assembly was determined. The primary factors that dictated the length were the total quantity of Oxone required for one batch and the convenience of handling. For ease of handling it was obvious that the shorter the assembly the better and over 12" length would not be recommended. The total Oxone required per change-out period (or batch) was dictated per the design requirements of a minimum of 5 grams of Oxone per liter of collected urine. For purposes of discussion in this report, one batch is defined as the total quantity of liquid collected to dissolve one prefilter/Oxone assembly. The total quantity of liquid collected relates to the use rate (number of astronauts on board), the change-out frequency (once or twice a day), and the quantity of flush water.

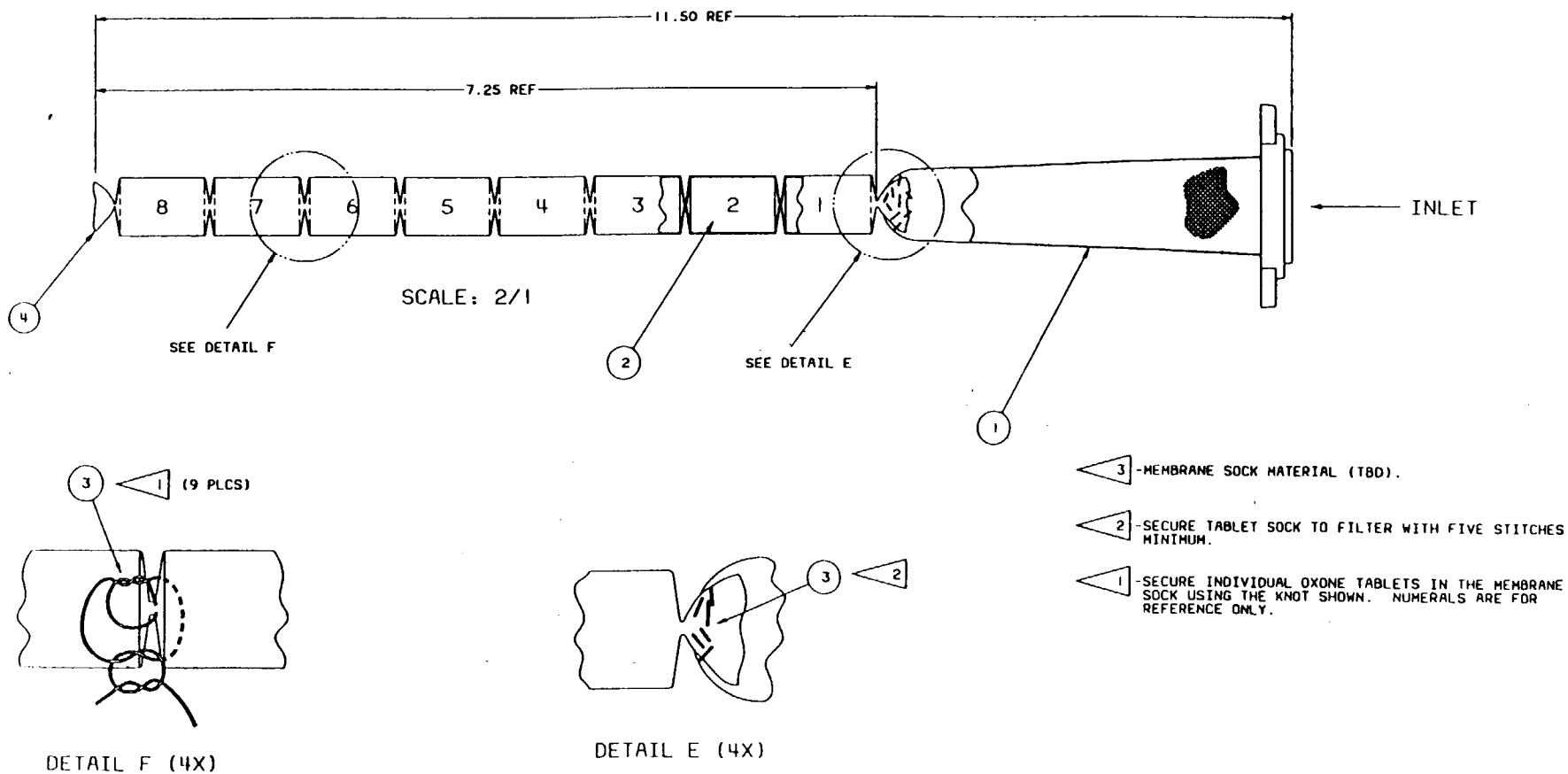


Figure 7

Layout of the Urine Pretreat/Prefilter Assembly that Contains Several Oxone Tablets in a Casing Attached to a "Wind Sock" Type Debris Prefilter

Based on the above variables, the total quantity of Oxone required per prefilter/Oxone assembly could range considerably. This is considered acceptable since the concept of individual Oxone tablets assembled into a casing allows a variable quantity of tablets to be used (i.e., 4 to 8 per assembly depending on the mission or use rate). During the test program each tablet contained about 6 grams of Oxone and binder. A total of 8 tablets were contained in each urine pretreat/prefilter assembly (see Figures 8 & 9). Therefore, each test assembly contained approximately 48 grams of Oxone and binder. Subtracting out the binder this would leave 46.3 grams of Oxone for pretreating 9.26 liters (20.6 lbs) of urine maximum at 5 grams of Oxone per liter of urine.

Oxone Table Dissolution Rate - Another key factor in definition of this concept for urine pretreat was establishing an acceptable and controllable dissolution rate of the Oxone tablet in a two-phase stream of urine and air. The starting point for this investigation was the previous effort under the baseline Space Station Commode/Urinal System (C/US) program which defined plasdone as a prime candidate binder. A binder is necessary to hold together the compressed Oxone powder in a tablet form and provide a slower and controlled dissolution rate for releasing the Oxone into the collected urine. The plasdone did slow down the dissolution rate of the tablet. However, it still allowed large pieces of the tablet to erode (or break) off. This condition was unpredictable and the smaller chunks caused a variation in the dissolution rate.

Polyethylene Glycol (PEG) was selected as an alternative binder to correct this condition. Various molecular weights, PEG to Oxone ratios, and mixing processes were investigated to provide an Oxone tablet that could be properly pressed and have the acceptable dissolution rate. After mixing and processing the Oxone powder and the PEG, approximately 6 grams of the material can be loaded into a 0.5 inch diameter cavity die and would be pressed into a tablet form. A Carver model "C" manual hydraulic press was used to provide a maximum load of 20,000 psi. The original Oxone powder is reduced in volume by a compaction ratio of 1.8:1. The final compressed size of a 6 gram tablet is 1.27 cm (0.5 inches) in diameter by 2.03 cm (0.8 inches) long.

A benchtop test rig was built to assist in obtaining a more controllable measure of dissolution rates while exposing a tablet or prefilter pretreat assembly of tablets to a more realistic or standard set of inlet conditions. The rig schematic is shown in Figure 10 and a photograph is shown in Figure 11. To obtain a baseline dissolution rate of the Oxone tablets during initial testing it was decided to use only water for which quantity, flowrate, and temperature could be easily controlled by this rig. An airflow rate of 10 cfm was provided to duplicate the airflow rate of the Space Station urinal. A clear length of 7/8" I.D. Lexon tubing was installed to simulate the urine collection hose and to visually inspect the liquid flow over the tablet (or prefilter assy). In addition, the tube was positioned vertically to minimize the effects of gravity during two-phase flow. (Refer to Figure 12).

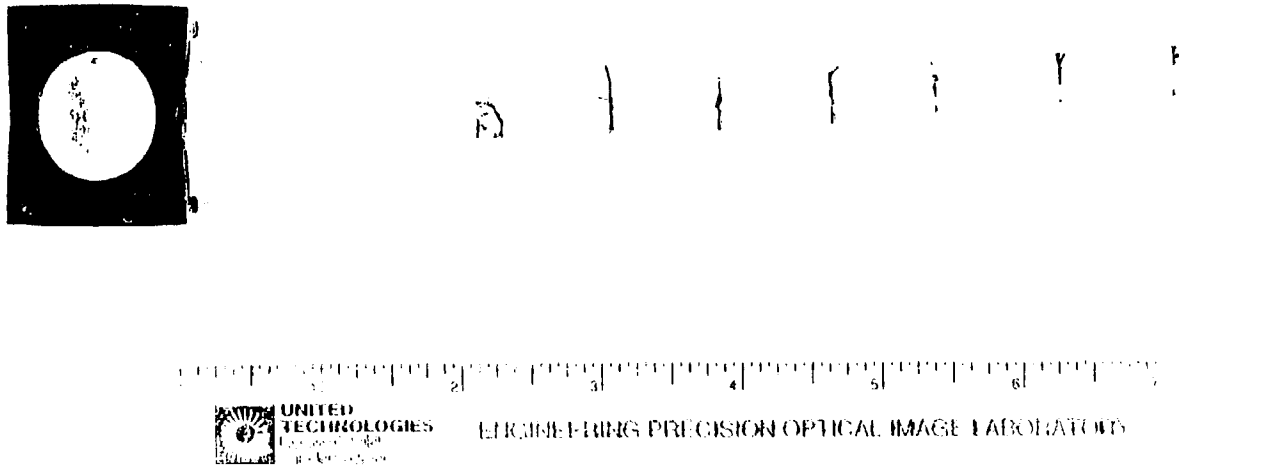


Figure 8

Photo of a Typical Urine Pretreat Prefilter with Eight Oxone Tablets and an Overall Length of Approximately 11.50 inches



Figure 9 Photo of Urine Pretreat Prefilter Showing Flexibility

MODEL		TITLE	PRETREAT EROSION RATE TEST SET UP	BY	DOM RETHME.
FILE				DATE	15 SEPT 94
JOB	CWF C10 000A			PAGE	2 OF 3

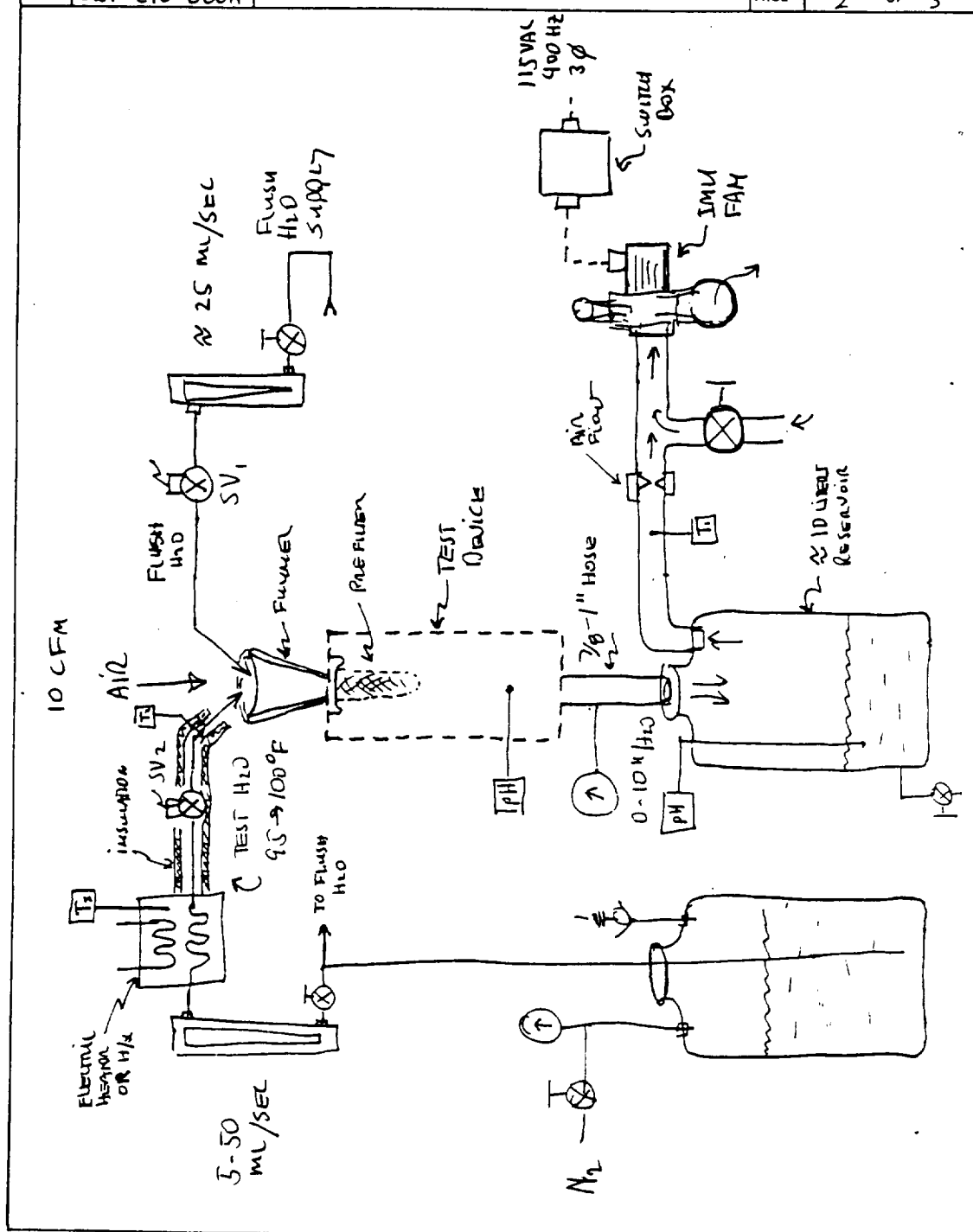


Figure 10 Schematic of Benchtop Dissolution Rate Rig

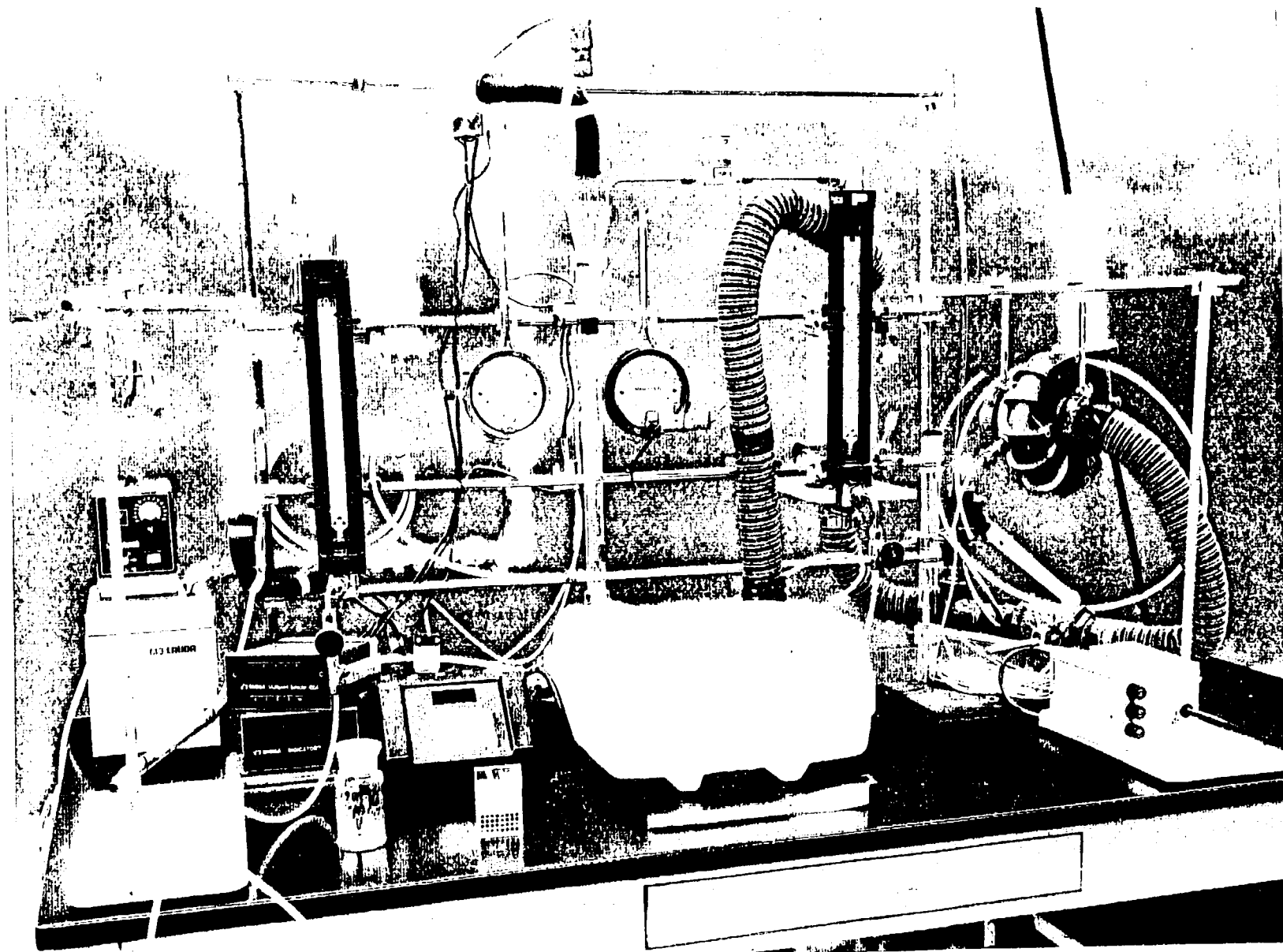


Figure 11

Photo of the Dissolution Rate Test Rig

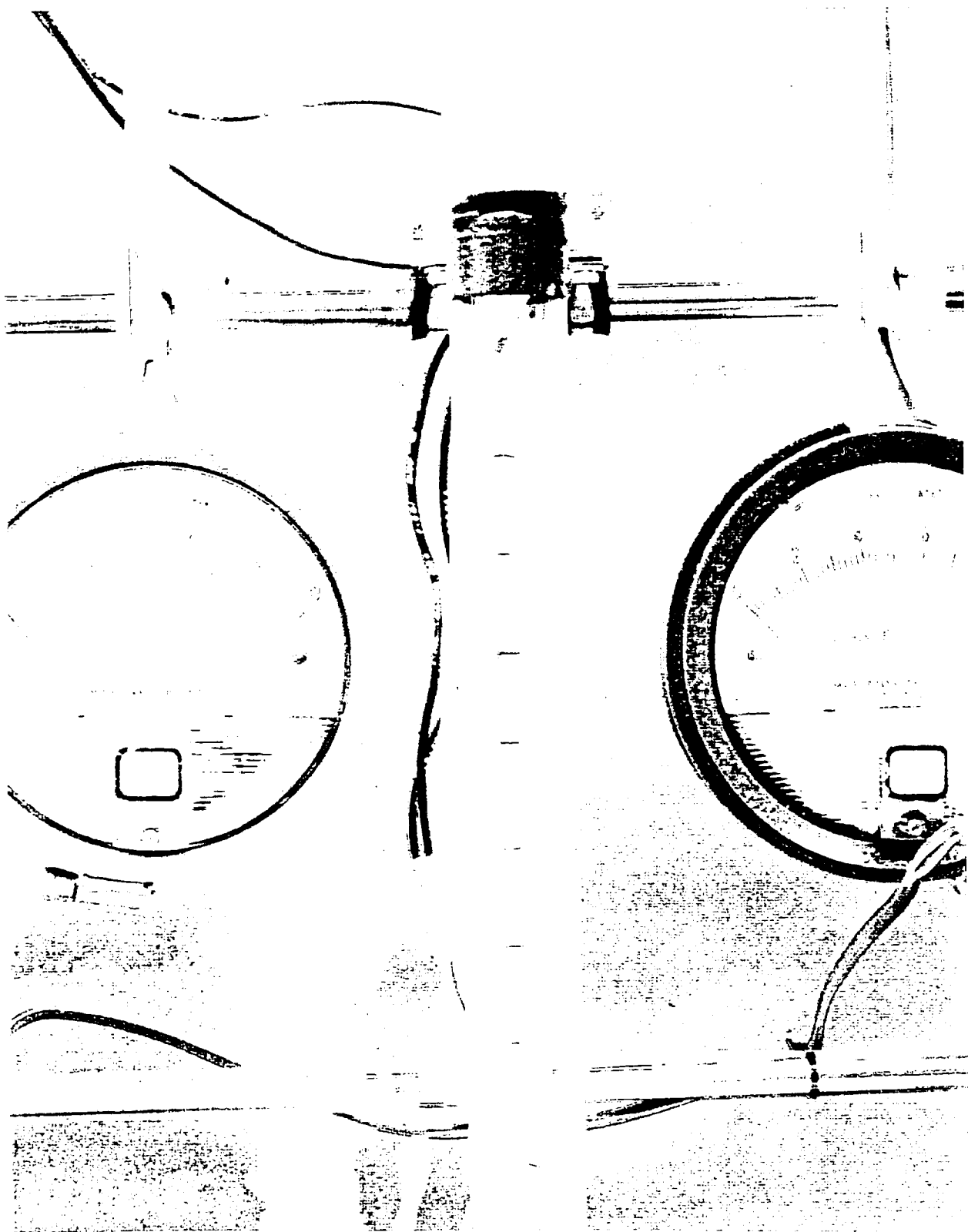


Figure 12 Closeup View Showing a Typical Urine Pretreat Prefilter Hanging in a Clear 7/8" I.D. Tube to Simulate the Urine Collection Hose

A series of tests were conducted using this rig which included the following: single tablet, multiple tablet, and ΔP tests.

Single Tablet Tests - Test conditions for the single tablet "quick" tests were as follows:

<u>Perimeter</u>	<u>Tablet #1</u>	<u>Tablet #2</u>
Initial Tablet Weight (g)	5.13	5.59
H ₂ O Flow Time (sec)	20	20
H ₂ O Flow Rate (ml/sec)	25	20
Air Flow Time (min)	3	3
Air Flow rate (CFM)	10	10
Flush H ₂ O Used	None	None

Figure 13 shows the tablet weight versus number of cycles plot of Tablet #1 and Tablet #2. The shape and slope of the curves are characteristic for tablet dissolution rate in a cyclic two-phase flow stream. The curve, for discussion purposes, is divided into three areas (or rates).

The first portion of the curve represents the initial wetting of the tablet with an associated smaller weight loss. The middle portion of the curve has a greater slope with a larger weight change between each cycle. The last portion of the curve has a lesser slope as the weight change starts to fall off. It was observed during this portion of the curve that the tablet size had been reduced significantly along with the surface area. Therefore, the cyclic weight reduction was less. To obtain this curve the tablet had to be removed after each cycle of input H₂O flow. During this test the input H₂O was also pretreated to approximate body temperature. The actual temperature recorded in the data sheets shown in Appendix E was the inlet peak temperature during the 20 sec flow time. The peak inlet temperature ranged from 98.2 to 104.4°F. During testing of Tablet #2 a pH test was conducted on the downstream flow during each flow cycle to obtain an instantaneous pH reading. To obtain this reading a pH indicator strip was inserted into the flow stream. As expected the pH was lower during the earlier cycles and as the tablet was reduced in size the pH was higher. The pH initially ranged about 3 1/2 to 4 and finally rose to 4 1/2 to 5. The reasons for conducting these single tablet tests were mostly exploratory and to check out the bench setup. However, the results indicated that the concept was working sufficiently to proceed to a prefilter/multiple Oxone tablet assembly.

Multiple Oxone Tablet Assembly Tests - At this point in the test program the fabrication of Oxone tablets and prefilter assembly had progressed enough to initiate testing at the multiple tablet level of assembly. Six (6) tablets were processed and pressed with 10% PEG. Each tablet weighed approximately 5 grams and the total tablet weight for the assembly was 31.66 grams (see data sheet in Appendix E). The

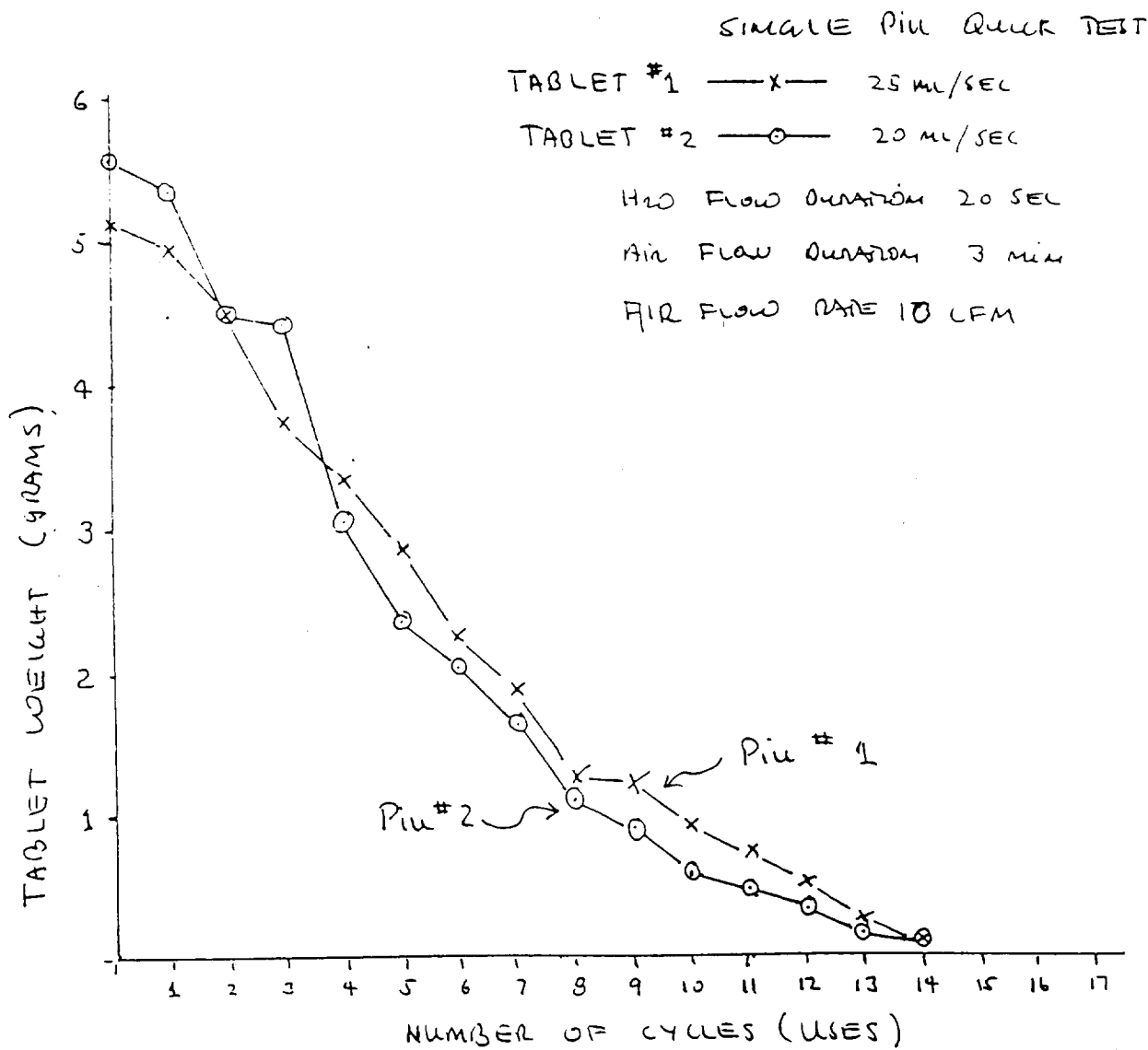


Figure 13

Test Results for the Dissolution Rate of Two (2)
Individual Oxone Tablets

test was conducted by inserting the prefilter assembly in the simulated urine collection hose as shown in Figure 12. Inlet test conditions were set up as follows:

H ₂ O Flow Time (sec)	20
H ₂ O Flow Rate (ml/sec)	20
Air Flow Time (min)	3
Air Flow Rate (CFM)	10
Cycle Duration (min)	5
Flush H ₂ O Used	None

Figure 14 shows a plot of total tablet weight versus cycles (or uses). Again the curve shows the characteristic shape of the initial wetting, the early steep slope, and the final portion where the dissolved quantity of tablet decreases per cycle. The test was stopped after 20 cycles were completed and the total residual undissolved weight of the tablets was 0.87 grams. Subtracting this from the initial weight and also subtracting 10% PEG resulted in 27.91 grams of Oxone dissolved. Over the 20 cycles a total throughput of 9.33 liters of H₂O was collected. The ratio of grams of Oxone to liters of water was 2.99, which was below the goal of 5 grams/liter. The low ratio of 2.99 grams/liter was considered to be within an acceptable range at this point in testing since heated water was used as a test fluid. A more realistic dissolution rate was expected with the use of fresh urine. The pH of the 9.33 liters of water in the collection reservoir was 3. The initial flowstream pH reading was 2 1/2 and after 20 cycles was at 4 to 4 1/2. It should be noted that the pH with water is expected to be slightly lower since urine will buffer the acidity of the Oxone.

Pressure Drop Tests - As previously mentioned there was a concern that the Oxone tablets hanging in the urine collection hose would cause a slight reduction in airflow because of the additional ΔP . The tare ΔP of a funnel and 7/8" ID Lexon tube was recorded at 0.8 inches of H₂O at 10 CFM. Adding the six (6) tablet urine pretreat prefilter increased the ΔP to 1.7 inches of H₂O. Therefore, the actual differential pressure for the prefilter assembly is 0.9" of H₂O which is still less than the specification limit of 1.0" of H₂O for the prefilter by itself. The new prefilter housing internal diameter was enlarged to a 1.0 inch diameter and with a matching larger prefilter surface area would offset some of the increased ΔP of the Oxone tablets. A new larger surface area prefilter was not available for testing at this time.

Prototype Test Verification - This portion of the urine pretreat injection test program includes several types of tests to verify the concept and prototype hardware. The tests to be discussed are:

- day long term urine collection
- Oxone concentration evaluation in urine

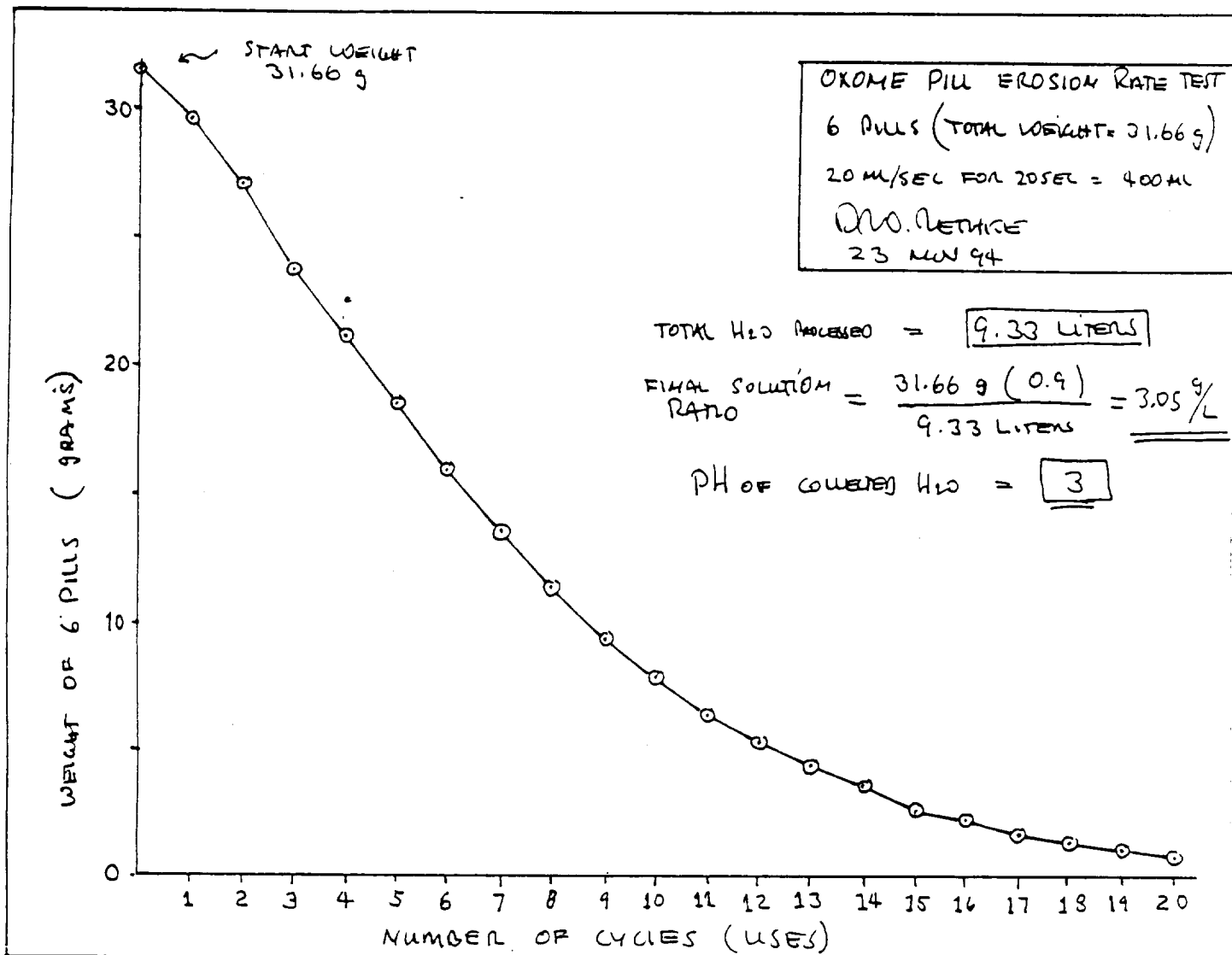


Figure 14

Test Results for the Dissolution Rate of a Six (6)
 Tablet Urine Pretreat Prefilter

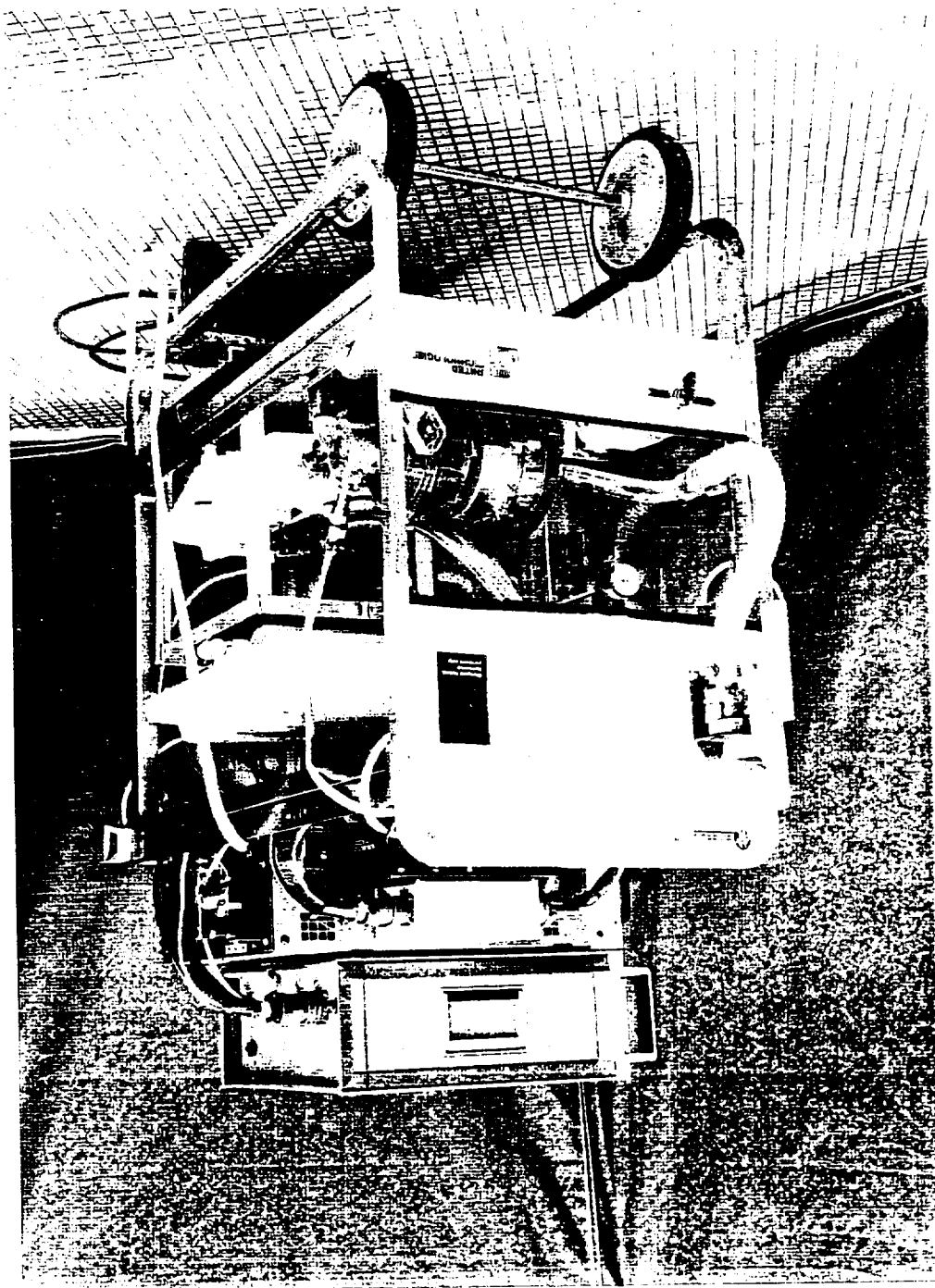
- microbial air sampling
- Oxone tablet storage stabilization

30-Day Long-Term Urine Collection Test - This test was the most significant test to verify the acceptability of this concept because it was conducted with fresh collected urine using actual concept pretreat hardware that was previously installed on the Shuttle EDO DTO urinal over a sufficient period of time. The test plan for the urine collection test is included in Appendix F. The portable test rig was fabricated using the Shuttle EDO DTO Urine Fan/Separator with clear Lexon housings. This Urine Fan/Separator, Hamilton Standard P/N SV 805378-1, was authorized to be used for this test program by Rockwell/NASA JSC and is the same hardware baselined for the Space Station Commode/Urinal System. Figure 2 of the test plan in Appendix F shows a schematic of the portable urine collection test rig and Figures 15 thru 17 show the rig prior to testing. For ease of operation by male volunteers the rig was modified to add a automatic "on"/"off" user lid, see Figures 18 & 19. Also, the Fan/Separator, itself, was lowered to allow the clear Lexon tube to be as vertical as possible for convenient use in a one-gravity ("terrestrial") mode.

On 12 February 95 a pretest baseline test was performed on the portable urine collection test rig to establish the operational characteristics of the Fan/Separator prior to exposure to long term urine and pretreat testing. This test was conducted using water as a test fluid. Test results are shown in Table A. Also, as part of the baseline test an eight (8) tablet urine pretreat/prefilter was subjected to a dissolution rate test using water in the benchtop dissolution rate test rig. For this test and the subsequent tests each urine pretreat/prefilter was assigned a serial number (i.e., S/N UPIS-XXXX) which would be associated with a specific batch of collected test media (i.e., BATCH # UPIS-XXXX). A plot of total tablet weight versus use cycles is shown in Figure 20.

The long-term urine collection test with pretreat was started on 14 February 95. Prior to the workday the portable urine collection test rig was positioned in a heavily traveled men's bathroom for "real time" urine collection and pretreatment by interested volunteers. A fresh urine pretreat/prefilter assembly (S/N UPIS-A001) was installed in the prefilter housing to collect the first batch. A stereo lithography (SLA) translucent prefilter housing identical to the titanium housing was used for viewing purposes. After about 4 1/2 hours 4 of the 7 tablets were totally dissolved and a data sheet was filled out for the first batch of collected and pretreated urine. The first of 40 data sheets is presented in Figure 21 for batch #UPIS-A001. After draining the collection tank (one batch) a new urine pretreat/prefilter was inserted for the next batch collection and data point. To obtain consistent data it was necessary to eliminate as many of the controllable test variables as possible. The following conditions were considered controllable variables:

- **Batch Start Time** - After the first several batches it was determined that the use rate (i.e., number of walk-in donors) would only support one batch a day.



Portable Urine Collection Test Rig (i.e., "Wizz on Wheels") Incorporating a Flight-Type Urine Collection Hose and EDO WCS Fan/Separator

Figure 15

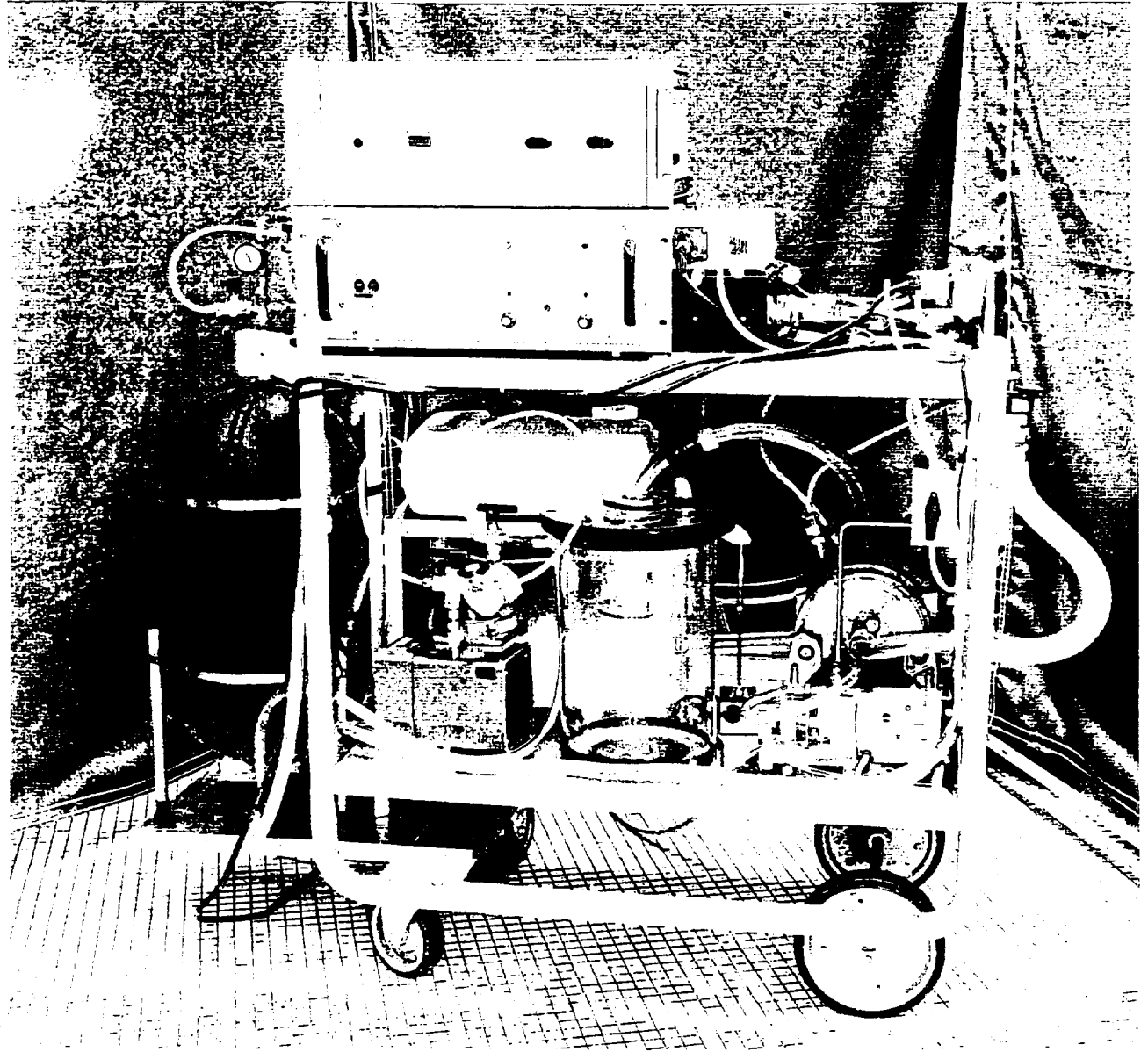


Figure 16

Side View of the Portable Urine Collection Test Rig
Showing the 120 VDC Power Supply, Control Box,
Flush H₂O Pump, and Clear Odor/Bacteria Filter Housing

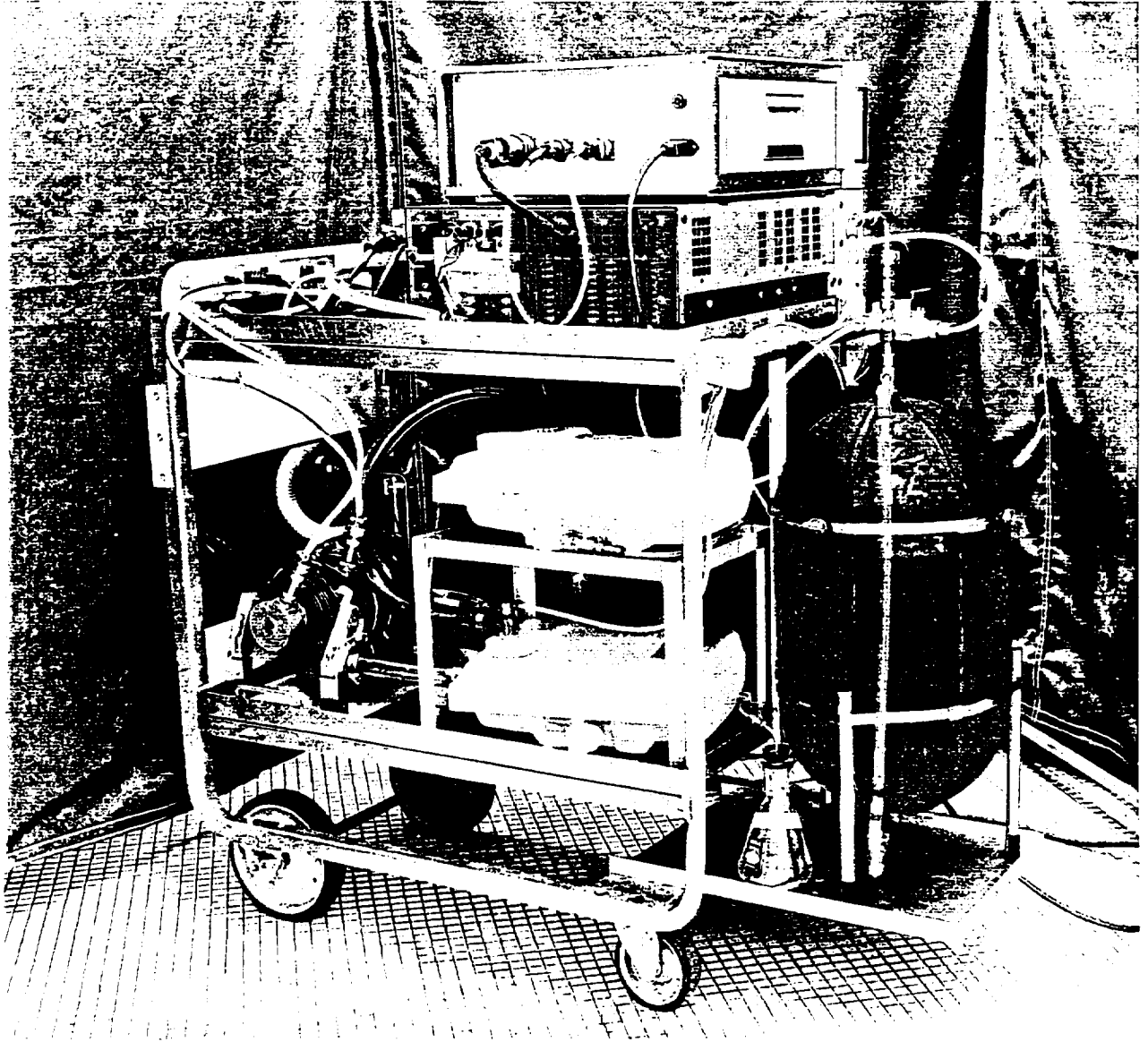


Figure 17

Side View of the Portable Urine Collection Test Rig
Showing the 35 Liter Pretreated Urine Reservoir

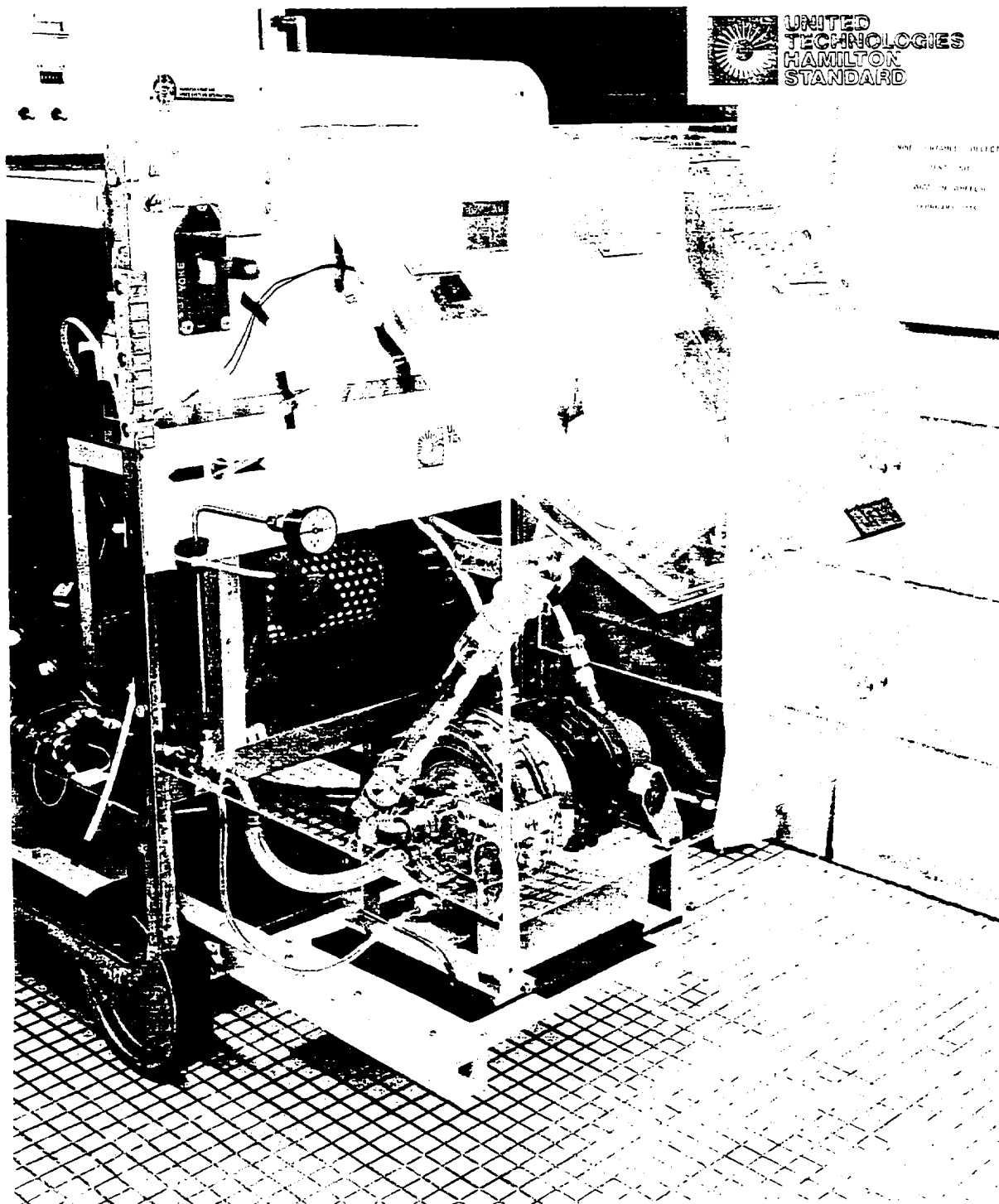


Figure 18 User Interface View of the Portable Urine Collection Test Rig Showing the Lowered Position of the Urine Fan/Separator and the Manual "Lift On"/"Close Off" User Control

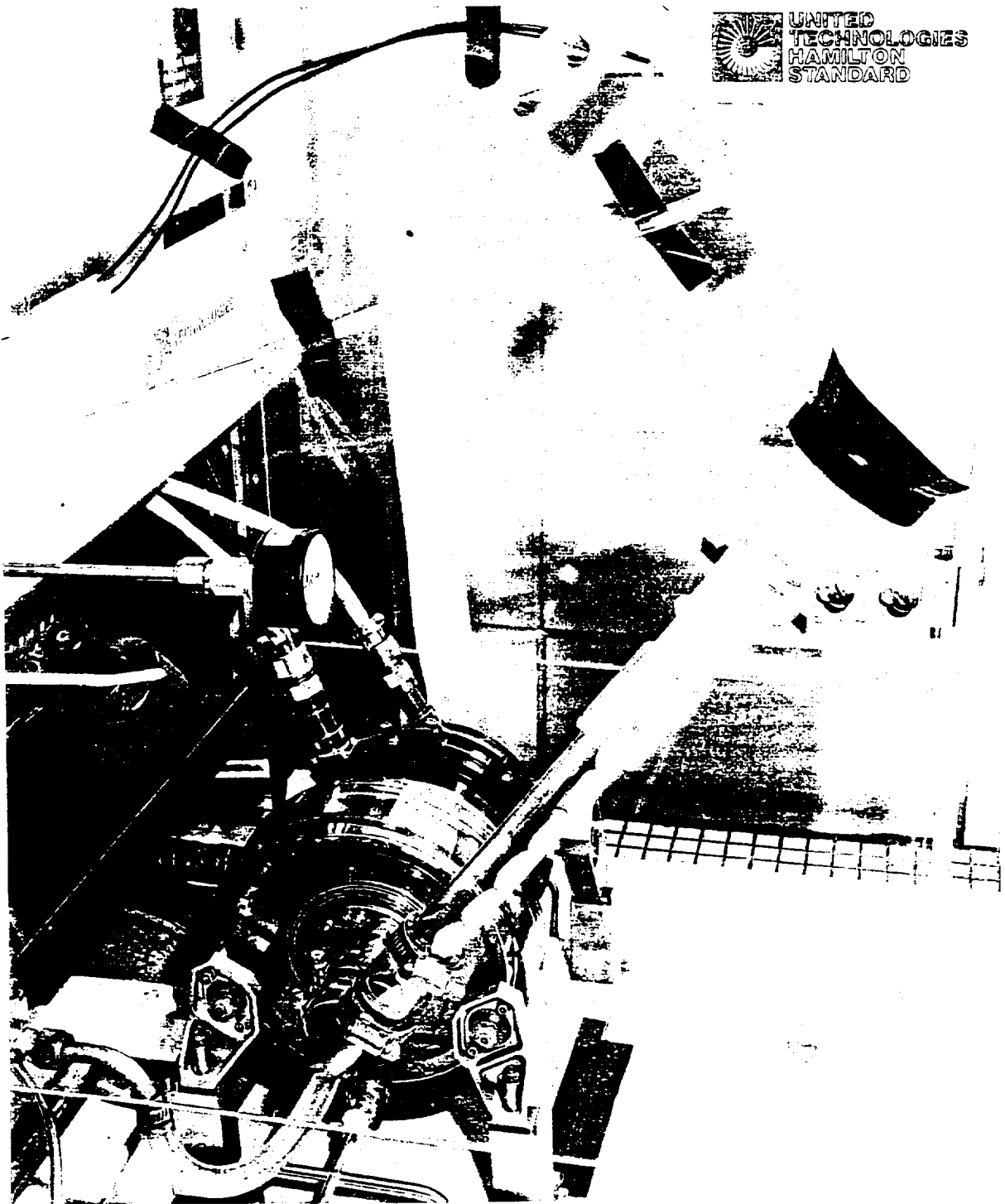


Figure 19

View Showing a Partially Dissolved Urine Pretreat
Prefilter Installed and the Clear Separator Housing

**Table A Portable Urine Collection Test Rig
Pre and Post Baseline Test Results**

<u>Condition/Parameter</u>	<u>Pretest</u>	<u>Post Test</u>
Date of Test	12 Feb 95	2 May 95
Pre Filter Used	Mockup Pretreat Filter Assy	Mockup Pretreat Filter Assy
Start/Start Cycle Readout (cycles)	91	1467
Test Rig Counter (cycles)	021678	023054
Separator Running Time (Hr)	2.3	54.5
Voltage Supply (VDC)	120 VDC	120 VDC
Total Current Fan/Sep (Amps)	0.85-0.90	0.88-0.90
Fan Current Steady State (Amps)	0.60	0.55
Fan Current Max Start Up (Amps)	0.8	0.8
Sep Current Steady State (Amps)	0.42	0.4
Sep Current Max Start Up (Amps)	1.2	1.2
Tank Pressure (psi)	8.0	8.0
Sep Outlet Pressure (psi)	10.0	9.0
Fan/Sep Post Use Run Time	1 min, 8 sec	1 min, 8 sec
Flush Time (sec)	29 sec	29 sec
Flush H ₂ O Qty (ml)	80 ml	79 ml
Post Test Flush of System	N/A	2 liters

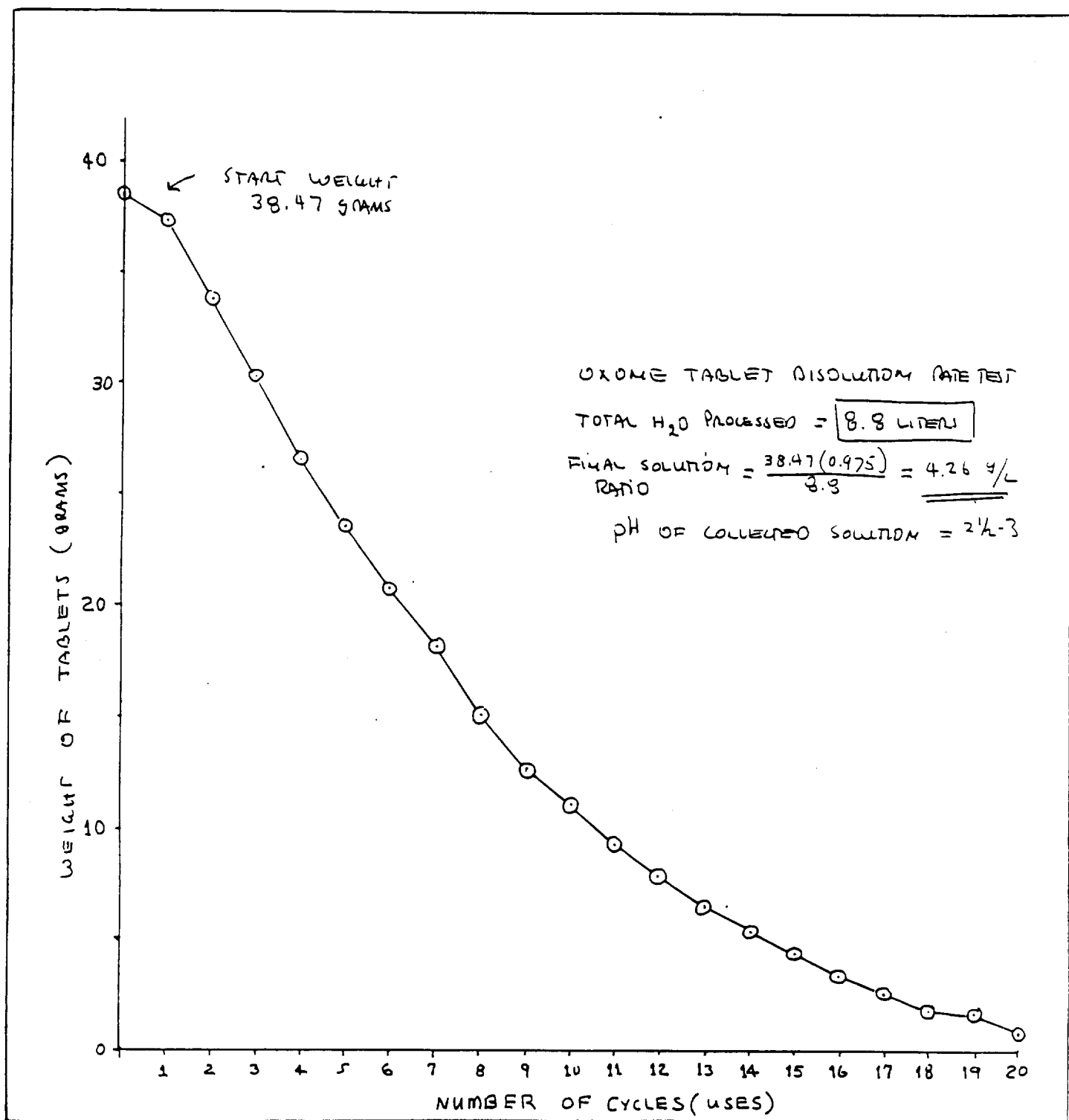


Figure 20

Dissolution Rate Curve for Pretest Baseline

MODEL		TITLE	BY
FILE		DATA SHEET	DATE
JOB			PAGE
			OF

UPIS 30 DAY EVALUATION TEST

BATCH # UPIS-A001 TUESDAY (AM)

PARAMETER	INITIAL	FINAL	Δ
TIME & DATE (—AM/PM —/—/—)	8:00AM 2/14/95	12:30PM 2/14/95	
S/N OF PRETREAT/PREFILTER INSTALLED	S/N A001	X	X
STOP/START CYCLE READOUT (#)	110	134	24
TEST RIG COUNTER (#)	~	021721	~
SEPARATOR RUNNING TIME (HOURS)	4.9	5.7	0.8
ACCUMULATOR PRESSURE (PSIG)	9.0	9.0	X
WEIGHT OF URINE COLLECTED (LBS)	0.0	15.8	15.8 LBS 7.170 L
PH OF URINE COLLECTED (PH)	X	3½ - 4	X
200 ML SAMPLE I.D. NUMBER (#)	X	SEE COMMENTS ①	X
NUMBER OF PILLS TOTALLY DISSOLVED	X	4 OF 7	X
WEIGHT OF PRETREAT (GRAMS)	44.14	44.09	< 0.05
VISUAL STATUS OF TRANSPARENT LINES	CLEAR (NEW)	CLEAR ②	NO CHANGE
OTHER NOTES & COMMENTS: ① DR FLUSH FLUSHED THE SAMPLE (BATCH) BY MISTAKE DOWN THE TUBES ② NOTICED FOAM IN URINE DOWN STREAM OF FAN/SEP			
FLUSH H ₂ O USED (ML)	$(24 \text{ CYCLES}) (80 \text{ ML/CYCLE}) = 1920 \text{ ML}$		
RATIO OF OXONE/URINE + FLUSH	$\frac{(44.09) \text{ g OXONE USED}}{(7.170) \text{ LITERS}} = 6.15 \text{ g/L}$		
AVERAGE VOID (ML/CYCLE)	$\frac{(7170 \text{ ML TOTAL COLLECTED}) - (1920 \text{ ML FLUSH})}{(24 \text{ CYCLES})} = 219 \text{ ML/CYCLE}$		

Figure 21

Typical Data Sheet Filled Out for Collection of One Batch of Pretreated Urine

Starting a collection batch at the beginning of a work day provided a relatively consistent donation population and the tablets would be dissolved around noon to mid-afternoon. This was also convenient for setting up before the day shift started and then taking data in the afternoon during the slow use time. This sequence also eliminated the variable that was noted for batch #UPIS-A002, which was started in the afternoon. After the day shift when the test was secured, there were still over 3 grams of Oxone left in the prefilter assy. For batch #UPIS-A004 the same mid-day collection start time condition occurred and it was decided to leave the undissolved tablet assembly in the test rig overnight (~16 hours without use) causing another variable.

- **Residual Undissolved Oxone** - The most opportune time in a urine batch collection process would be to stop collection is the point when the last tablet(s) dissolved into the flow stream. This was not totally practical since this was a visual observation and somewhat objective as to when the last granule of Oxone dissolved. If the tablet assembly was removed too soon the calculated ratio would be too high. If the tablet assembly was left in the rig excessively long after completely dissolving the dissolution rate calculation would be inaccurate and also allow some untreated urine thru the separator. Therefore, towards the end of the batch cycle as the tablets dissolved the rig would be checked routinely to visually determine the best time to take data points.

The urine collection test was conducted over an eleven (11) week time period for a total of 40 batches collected with the last batch collected on 28 April 95. Table B is a summary of the data collected from the daily data sheets. The following are explanations of each column of the daily summary data sheet:

- **Batch I.D. number** - This is the identification of the batch of urine collected from UPIS-A001 thru UPIS-A040 for a total of 40 batches.
- **Use Cycles per Batch** - This is a count of the total number of times that the rig was cycled. This is assuming that nobody played with the lid causing extra cycles. Conversely, fewer cycles would have been recorded if the unit was used sequentially by two people without shutting the lid. It is believed that these readings are reasonably accurate. Therefore, the data was used to obtain the quantity of flush water processed. Cycles ranged from a low of 16 to a maximum of 42 with an average of 32. A total of 1276 use cycles was accumulated during the test.
- **Qty Liquid per Batch** - This was the total quantity of liquid collected in the reservoir of the portable collection rig per batch. This included 80 ml of flush H₂O that was collected automatically after each use. An average of 11.09 liters of liquid was collected per batch.

Table B Summary of Initial Data From UPIS Data Sheets

BATCH ID NO	USE CYCLES PER BATCH (NO.)	QTY LIQUID PER BATCH (mL)	WEIGHT OF PRETREAT USED (g)	PRETREAT TO LIQUID RATIO (g/Liters)	RESIDUAL PRETREAT (g)	ACIDITY OF BATCH (pH)	BATCH OXIDATION VALUE (%)	AVERAGE VOID LESS FLUSH (mL)	SEPARATOR RUNNING TIME (hr)	AVERAGE RUNNING TIME (MIN/CYCLE)
UPIS-A001	24	7170	44.09	6.15	<0.05	3 1/2-4	-	219	0.8	2.00
UPIS-A002	16	5440	38.24	7.03	3.07	4	0.0260%	260	0.6	2.25
UPIS-A003	25	8800	46.02	5.23	0.63	4 1/2	0.0200%	272	0.9	2.16
UPIS-A004	24	6710	47.94	7.14	0.00	4	0.0400%	200	0.9	2.25
UPIS-A005	25	7440	46.46	6.25	-0.20	4	0.0320%	218	0.8	1.92
UPIS-A006	34	11340	47.42	4.18	0.00	4 1/2	0.0130%	254	1.3	2.29
UPIS-A007	27	9800	48.11	4.91	-0.50	4	0.0320%	283	0.9	2.00
UPIS-A008	27	8440	41.90	4.97	0.66	4 1/2	0.0095%	232	0.9	2.00
UPIS-A009	32	12070	45.88	3.80	<0.10	4 1/2	0.0036%	297	1.2	2.25
UPIS-A010	30	12610	45.61	3.62	<0.10	5	0.0097%	340	1.2	2.40
UPIS-A011	37	15150	46.95	3.10	-0.20	5	0.0059%	329	1.4	2.27
UPIS-A012	31	11340	47.45	4.18	<0.10	4 1/2	0.0170%	286	1.1	2.13
UPIS-A013	32	11160	48.08	4.31	<0.10	4 1/2	0.0160%	269	1.1	2.06
UPIS-A014	31	10800	47.07	4.36	-0.20	4 1/2	0.0214%	268	1.1	2.13
UPIS-A015	35	10030	47.75	4.76	0.39	4 1/2	0.0085%	206	1.2	2.06
UPIS-A016	29	11160	45.02	4.03	0.00	4 1/2	0.0250%	304	1.0	2.07
UPIS-A017	31	11880	47.85	4.03	<0.05	5	0.0130%	303	1.2	2.32
UPIS-A018	34	12880	47.97	3.72	<0.05	4 1/2	0.0062%	299	1.3	2.29
UPIS-A019	34	12520	48.09	3.84	<0.05	4 1/2	0.0130%	288	1.3	2.29
UPIS-A020	31	10020	47.89	4.78	0.27	4 1/2	0.0150%	243	1.1	2.13
UPIS-A021	33	11700	48.11	4.11	<0.10	4 1/2	0.0180%	275	1.1	2.00
UPIS-A022	37	12340	48.09	3.90	0.13	5	0.0099%	253	0.4	0.65
UPIS-A023	30	10400	48.30	4.60	<0.05	4 1/2	0.0120%	268	1.0	2.00
UPIS-A024	42	12160	48.18	3.96	0.00	5	0.0044%	209	1.6	2.29
UPIS-A025	33	8980	47.73	5.31	0.55	4 1/2	0.0057%	192	1.1	2.00
UPIS-A026	35	12880	48.12	3.74	<0.05	4 1/2	0.0067%	288	1.2	2.06
UPIS-A027	33	12880	48.24	3.75	0.00	4 1/2	0.0053%	310	1.1	2.00
UPIS-A028	27	9620	47.97	4.99	0.25	4	0.0320%	276	0.9	2.00
UPIS-A029	36	11520	47.35	4.11	1.02	4 1/2	~	240	1.2	2.00
UPIS-A030	41	13790	47.66	3.46	0.00	5	~	256	1.5	2.20
UPIS-A031	31	11520	48.04	4.17	<0.30	4 1/2	~	292	1.3	2.52
UPIS-A032	37	12340	48.16	3.90	0.21	4 1/2	~	253	1.3	2.11
UPIS-A033	34	11790	48.20	4.09	-0.10	4 1/2	~	267	1.3	2.29
UPIS-A034	34	12340	48.02	3.89	-0.25	5	~	283	1.2	2.12
UPIS-A035	36	12700	48.04	3.78	>0.10	4 1/2	~	273	1.2	2.00
UPIS-A036	29	11100	48.19	4.34	0.00	4 1/2	~	303	1.0	2.07
UPIS-A037	33	11000	48.22	4.38	0.00	4	~	253	1.1	2.00
UPIS-A038	33	12800	47.74	3.73	0.38	5	~	308	1.1	2.00
UPIS-A039	36	13610	48.15	3.54	-0.10	5	~	298	1.7	2.83
UPIS-A040	37	11800	48.08	4.08	-0.15	4 1/2	~	239	1.3	2.11
TOTALS	1276	444030	1886.38	~	4.16	~	~	~	44.90	~
Averages	32	11090	47.14	4.54	0.22	4.54	0.0156%	267	1.1	2.10

- **Weight of Pretreat Used** - This is the total weight of the tablets used in grams which includes the binder. During the fabrication of the urine pretreat/prefilter each assembly was serialized and the total weight of the Oxone tablets including binder was recorded. The average total weight of eight (8) tablets in a prefilter assembly was 47.14 grams.
- **Residual Pretreat** - This was the total weight in grams of the undissolved tablets. As previously discussed an attempt was made to stop urine collection for each batch as the tablets were just about totally dissolved or shortly thereafter. When the used prefilter assembly was removed, it was visually inspected and the number of undissolved tablets would be recorded on the data sheet. Also, all of the undissolved chips would be cut out of the prefilter casing, weighed, and recorded.
- **Acidity of the Batch** - This is the pH reading taken on each batch using a litmus strip indicator. The procedure used was to drain about 1/2 of the rig collection reservoir. At this point the pH strip was held in the flow stream to get a reading. A second strip was used to back up the reading. The range of pH readings was usually between 4 to 5 with an average of 4 1/2 over 40 batches.
- **Batch Oxidation Value** - This is the percent of active oxygen in the pretreated collected urine. After each batch was collected, a 180 ml sample was drawn from the rig reservoir and analyzed. The method used was the standard iodometric titration recommended by DuPont in the Oxone data sheet. The Oxone data sheet is included in Appendix C.
- **Average Void Less Flush** - This is a calculated value of the average quantity of void in millimeters per each batch. This void volume was obtained by subtracting the 80 ml of flush water per cycle from the total liquid collected and dividing by the number of cycles per batch. The average void volume for the 40 batches was 267 ml.
- **Separator Running Time** - This is the total of time in hours for the Urine Fan/Separator as recorded by an elapsed time indicator. The total separator operating time for the complete test was about 45 hours which included at least 1276 on/off cycles.
- **Average Running Time** - This is a calculated time in minutes that the Fan/Separator would be running for each use (or void). The average on time is 2.1 minutes which includes the post use time delay shutdown period of 45 sec.

Table C includes a continuation of the data recorded and calculated from the daily data sheets. The following are explanations of each column of the calculated data sheet:

- **Batch I.D. No.** - same as Table B.
- **Weight of pretreat and binder** - same as Table B.
- **Percentage of binder**. This is the nominal percentage by weight used to process the individual Oxone tablets.
- **Weight of pretreat only** - This is the total weight of the tablets in grams with the appropriate percentage of binder subtracted out. The average weight of Oxone available per prefilter/pretreat assembly was 45.55 grams.
- **Qty of Liquid per Batch** - Same as Table B.
- **Qty of Flush Per Batch** - This is the total calculated volume in millimeters of flush water used in each batch. The volume was obtained by multiplying the void cycles by 80 ml per void.
- **Qty of Urine per Batch** - This is the total calculated volume in milliliters of urine per batch less the flush H₂O. The average volume of urine collected per batch was 8.55 liters.
- **Ratio of Oxone to Urine** - This is the calculated ratio in grams of Oxone per liter of urine. The goals of the test program was to demonstrate a proper pretreat ratio of at least 5. grams/liter.
- **Total Qty of Urine Collected** - This is a cumulative record of the urine collected for the 40 batches. The total volume of urine pretreated and collected was 342 liters.

Figure 22 is a plot of the Oxone to urine ratio for the total of 40 batches (or "data points"). The primary goal was to have the batch concentration ratio of Oxone to urine be just slightly higher than 5.0 grams/liter. It was assumed that there would be several uncontrolled variables that would cause the ratio to vary slightly from batch to batch. It was also assumed that the dissolution rate with fresh urine would have to be modified slightly to "zero in" on the 5.0 grams/liter goal. As demonstrated by the plot, the first few data points grounded around 8 grams/liter. Since the trend was on the high side the dissolution rate had to be slowed down slightly by changing the percentage of PEG from approximately 2.5% to 3.5%. This was a best estimate type of change but the results indicated that the trend in Oxone to urine ratio was reduced with the higher PEG percentages. Again several batches were observed and the average ratio did fall to an acceptable level. No further changes in the Oxone tablet chemical composition and

Table C Summary of UPIS Calculated Data for Urine Pretreat Test

BATCH ID NO	WEIGHT OF PRETREAT & BINDER (g)	PERCENTAGE OF BINDER (%)	WEIGHT OF PRETREAT ONLY (g)	QTY OF LIQUID PER BATCH (mL)	QTY OF FLUSH PER BATCH (mL)	QTY OF URINE PER BATCH (mL)	RATIO OF OXONE TO URINE (g/liter)	TOTAL QTY OF URINE COLLECTED (Liters)
UPIS-A001	44.09	2.5%	42.99	7170	1920	5250	8.19	5.25
UPIS-A002	38.24	2.5%	37.28	5440	1280	4160	8.96	9.41
UPIS-A003	46.02	2.5%	44.87	8800	2000	6800	6.60	16.21
UPIS-A004	47.94	2.5%	46.74	6710	1920	4790	9.76	21.00
UPIS-A005	46.46	3.5%	44.83	7440	2000	5440	8.24	26.44
UPIS-A006	47.42	3.5%	45.76	11340	2720	8620	5.31	35.06
UPIS-A007	48.11	3.5%	46.43	9800	2160	7640	6.08	42.70
UPIS-A008	41.90	3.5%	40.43	8440	2160	6280	6.44	48.98
UPIS-A009	45.88	3.5%	44.27	12070	2560	9510	4.66	58.49
UPIS-A010	45.61	3.5%	44.01	12610	2400	10210	4.31	68.70
UPIS-A011	46.95	3.5%	45.31	15150	2960	12190	3.72	80.89
UPIS-A012	47.45	3.5%	45.79	11340	2480	8860	5.17	89.75
UPIS-A013	48.08	3.5%	46.40	11160	2560	8600	5.40	98.35
UPIS-A014	47.07	3.5%	45.42	10800	2480	8320	5.46	106.67
UPIS-A015	47.75	3.5%	46.08	10030	2800	7230	6.37	113.90
UPIS-A016	45.02	3.5%	43.44	11160	2320	8840	4.91	122.74
UPIS-A017	47.85	3.5%	46.18	11880	2480	9400	4.91	132.14
UPIS-A018	47.97	3.5%	46.29	12880	2720	10160	4.56	142.30
UPIS-A019	48.09	3.5%	46.41	12520	2720	9800	4.74	152.10
UPIS-A020	47.89	3.5%	46.21	10020	2480	7540	6.13	159.64
UPIS-A021	48.11	3.5%	46.43	11700	2640	9060	5.12	168.70
UPIS-A022	48.09	3.5%	46.41	12340	2960	9380	4.95	178.08
UPIS-A023	48.30	3.5%	46.61	10400	2400	8000	5.83	186.08
UPIS-A024	48.18	3.5%	46.49	12160	3360	8800	5.28	194.88
UPIS-A025	47.73	3.5%	46.06	8980	2640	6340	7.26	201.22
UPIS-A026	48.12	3.5%	46.44	12880	2800	10080	4.61	211.30
UPIS-A027	48.24	3.5%	46.55	12880	2640	10240	4.55	221.54
UPIS-A028	47.97	3.5%	46.29	9620	2160	7460	6.21	229.00
UPIS-A029	47.35	3.5%	45.69	11520	2880	8640	5.29	237.64
UPIS-A030	47.66	3.5%	45.99	13790	3280	10510	4.38	248.15
UPIS-A031	48.04	3.5%	46.36	11520	2480	9040	5.13	257.19
UPIS-A032	48.16	3.5%	46.47	12340	2960	9380	4.95	266.57
UPIS-A033	48.20	3.5%	46.51	11790	2720	9070	5.13	275.64
UPIS-A034	48.02	3.5%	46.34	12340	2720	9620	4.82	285.26
UPIS-A035	48.04	3.5%	46.36	12700	2880	9820	4.72	295.08
UPIS-A036	48.19	3.5%	46.50	11100	2320	8780	5.30	303.86
UPIS-A037	48.22	3.5%	46.53	11000	2640	8360	5.57	312.22
UPIS-A038	47.74	3.5%	46.07	12800	2640	10160	4.53	322.38
UPIS-A039	48.15	3.5%	46.46	13610	2880	10730	4.33	333.11
UPIS-A040	48.08	3.5%	46.40	11800	2960	8840	5.25	341.95
Avg of 1-40	47.16	3.4%	45.55	11101	2552	8549	5.58	8.5
Avg of 1-5	44.55	2.7%	43.34	7112	1824	5288	8.35	5.3
Avg of 6-40	47.50	3.5%	45.83	11608	2640	8968	5.20	9.0
TOTALS OF 1-40	1886.38	~	1822.12	444030	102080	341950	~	341.95

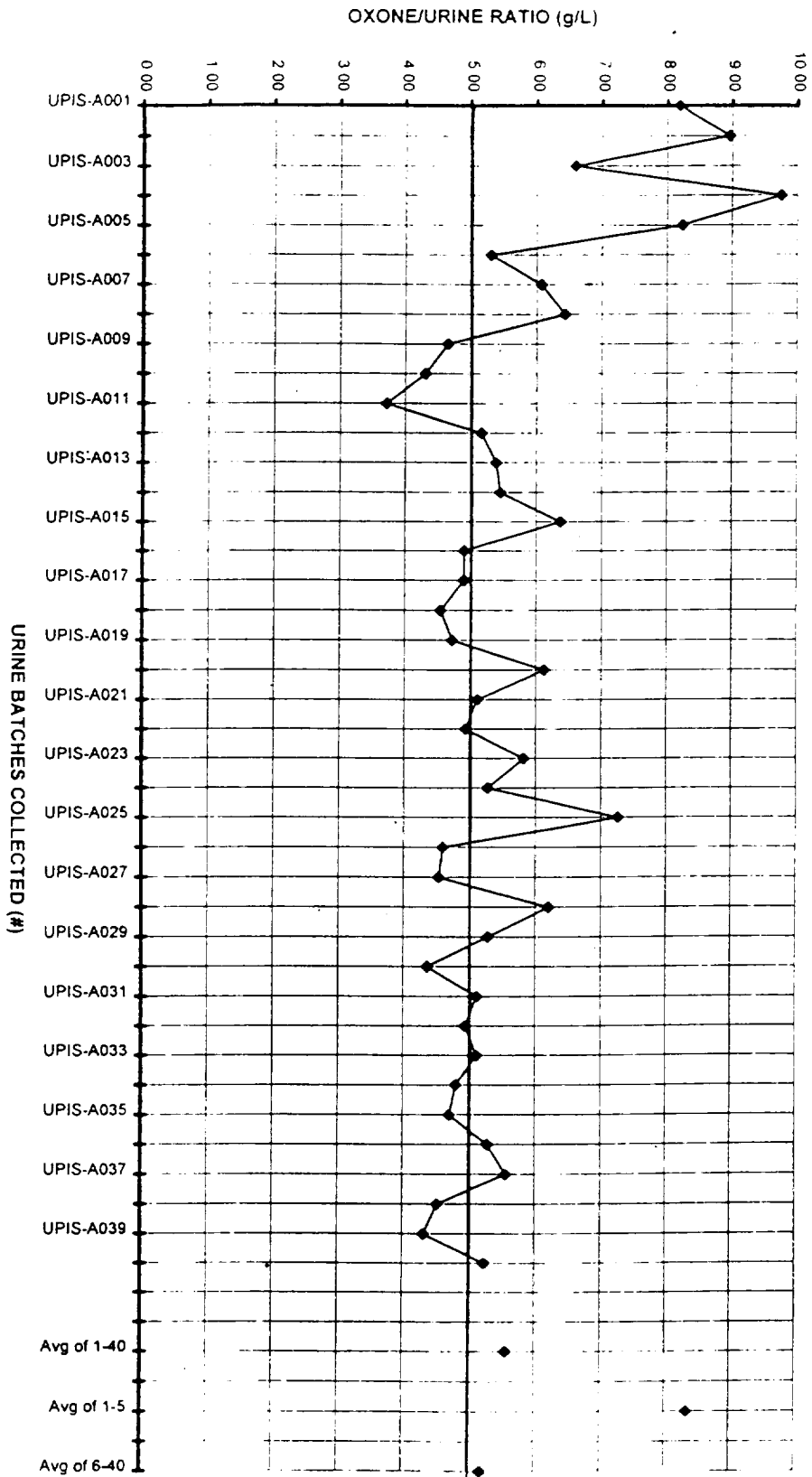


Figure 22
Long Term Urine Collection Test Ratio of Oxone to
Urine (g/liter) vs. Batch Collected

processing were made. The final average for batches UPIS-A006 thru UPIS-A040 was 5.2 grams of Oxone per liter of collected urine.

On 28 April 1995 the last batch was collected for a total quantity of urine processed at 341.95 liters (754 lbs). After the last batch was collected a post test baseline check was conducted on the fan/separator. Test results are shown in Table A and compared with the pretest results indicate that there was no performance degradation in the operation of the separator.

After completion of all testing the portable urine collection test rig was partially disassembled for post test evaluation of the Urine Fan/Separator and plumbing. A teardown of the Fan/Separator was conducted to visually examine the areas that were exposed to the collected urine. Figures 23 and 24 show some of the detail subassemblies and internal parts of the separator with no evidence of urine deposits. The same was true for the downstream dual in-line check valve teardown shown in Figures 25 and 26. Figure 27 shows other miscellaneous parts that were exposed to the urine, i.e., Lexon inlet tube, 90° stainless steel inlet elbow, 3/8" outlet tygon tubing, and outlet quick disconnects with tee. Refer to Figure 1 and 2 for a comparison of urine fan/seperator details which were not pretreated with Oxone. These details were exposed to only one flight for approximately 100 Kg (220 lbs) of urine.

A closer look at the outer o-seal area of the rotating Titanium drum showed some evidence of foreign material. These small particles were driven into the inboard side of the o-seal area by the centrifugal force of the rotating drum. Figures 28, 29, and 30 are magnified views of the debris which is considered to be small enough in size to have entered through the pores of the inlet prefilter. The small fibrous white material (or lint) was drawn into the separator by air entrainment of which most could be captured in the prefilter. The small red material (or chips) are from the reworked prefilter seal area which is a red silicone elastomer. It is not expected that these chips would be present on a flight quality pretreat/prefilter assembly. Refer to Figure 6, which shows a typical test prefilter that was cut down for use in the urine collection test.

Oxone Concentration Evaluation in Urine - During urine pretreat test program it was considered necessary to determine the minimum Oxone concentration level that would provide control of microbial growth in urine. A separate quantity of urine was collected in a container without pretreat and provided to the Microbiology Lab for evaluation. Data indicated that for Oxone concentrations as low as 4.0 grams/liter provide excellent microbial growth control and; levels down to 3.0 gram/liter still provide partial control in collected urine at room temperature. The complete test and evaluation is contained in Appendix G.

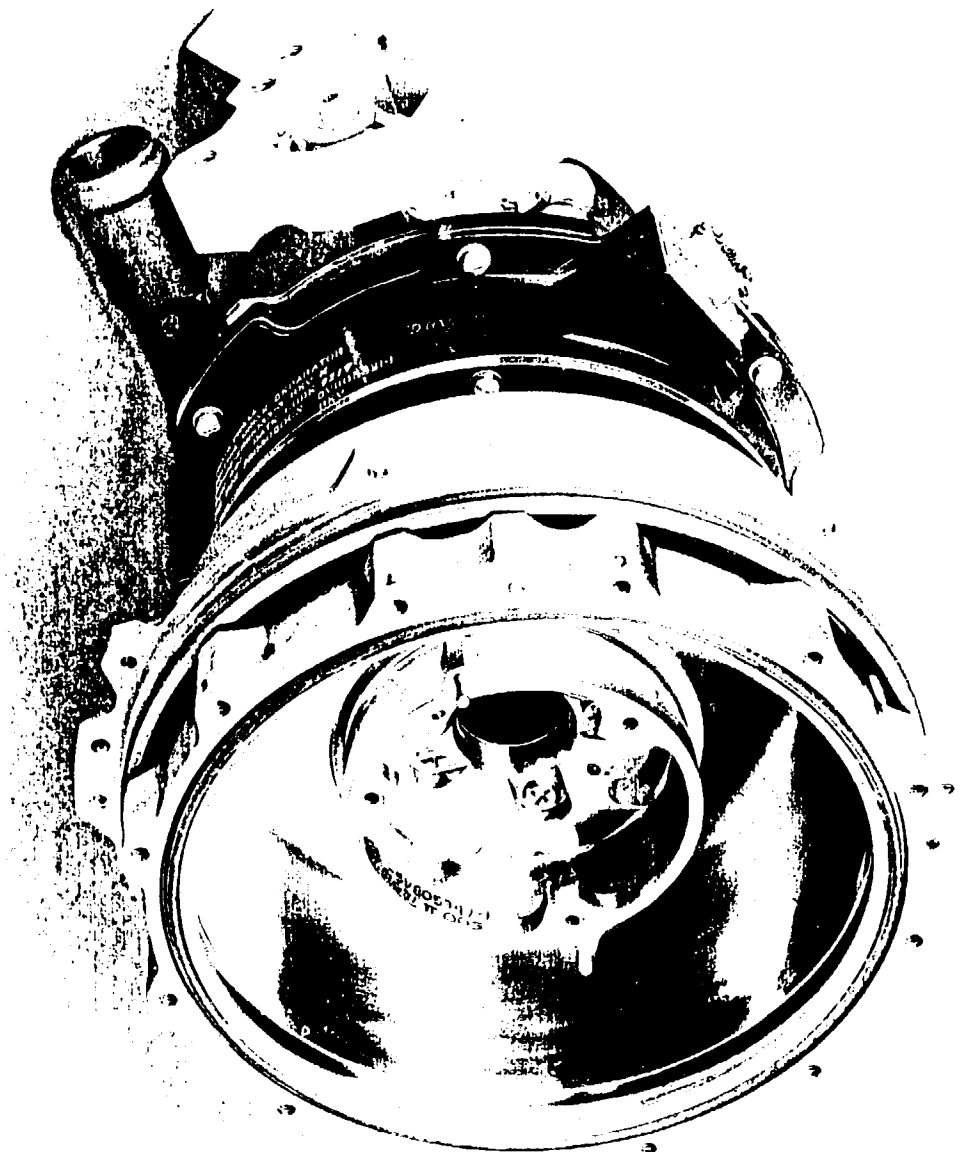


Figure 23

Post Test Teardown of the Urine Separator Showing
Clean Internal Surfaces of the Rotating Drum



Figure 24

Post Test Teardown of the Urine Separator Showing the Inlet Housing and Pitot Tube with No Urine Deposits

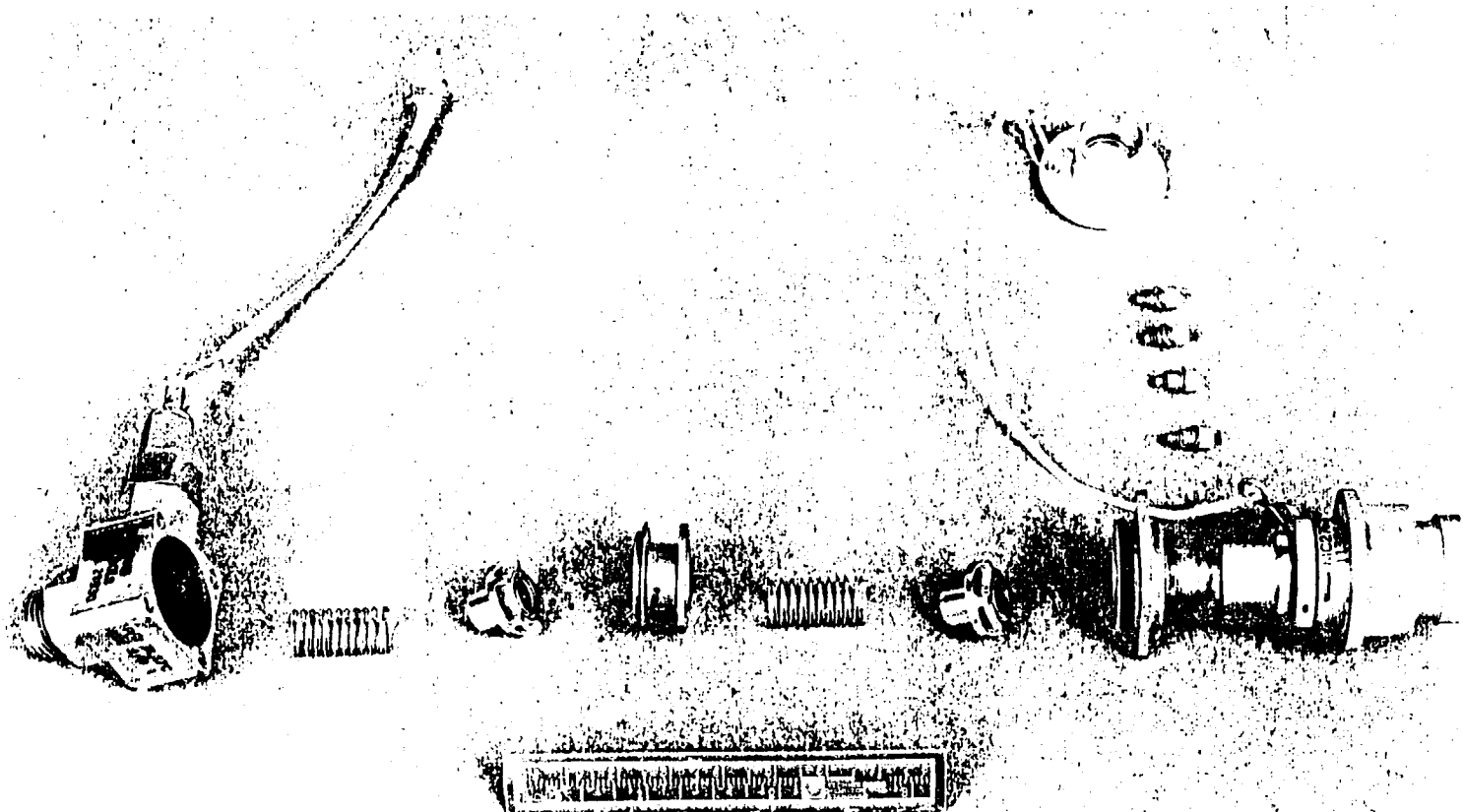


Figure 25

Post Test Teardown of the Dual In-line Check Valve With
No Evidence of Foreign Debris or Urine Deposits

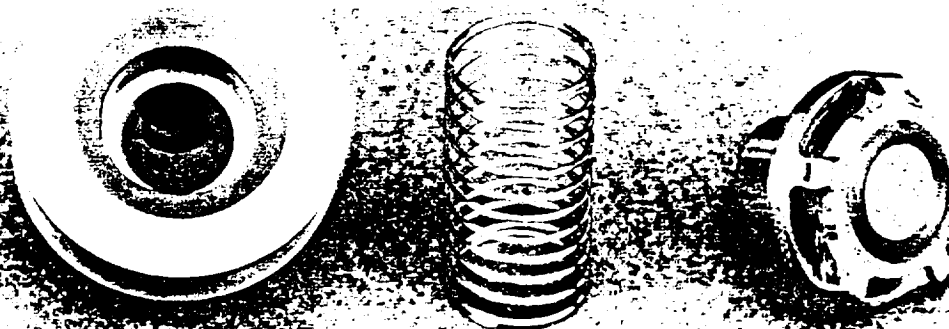
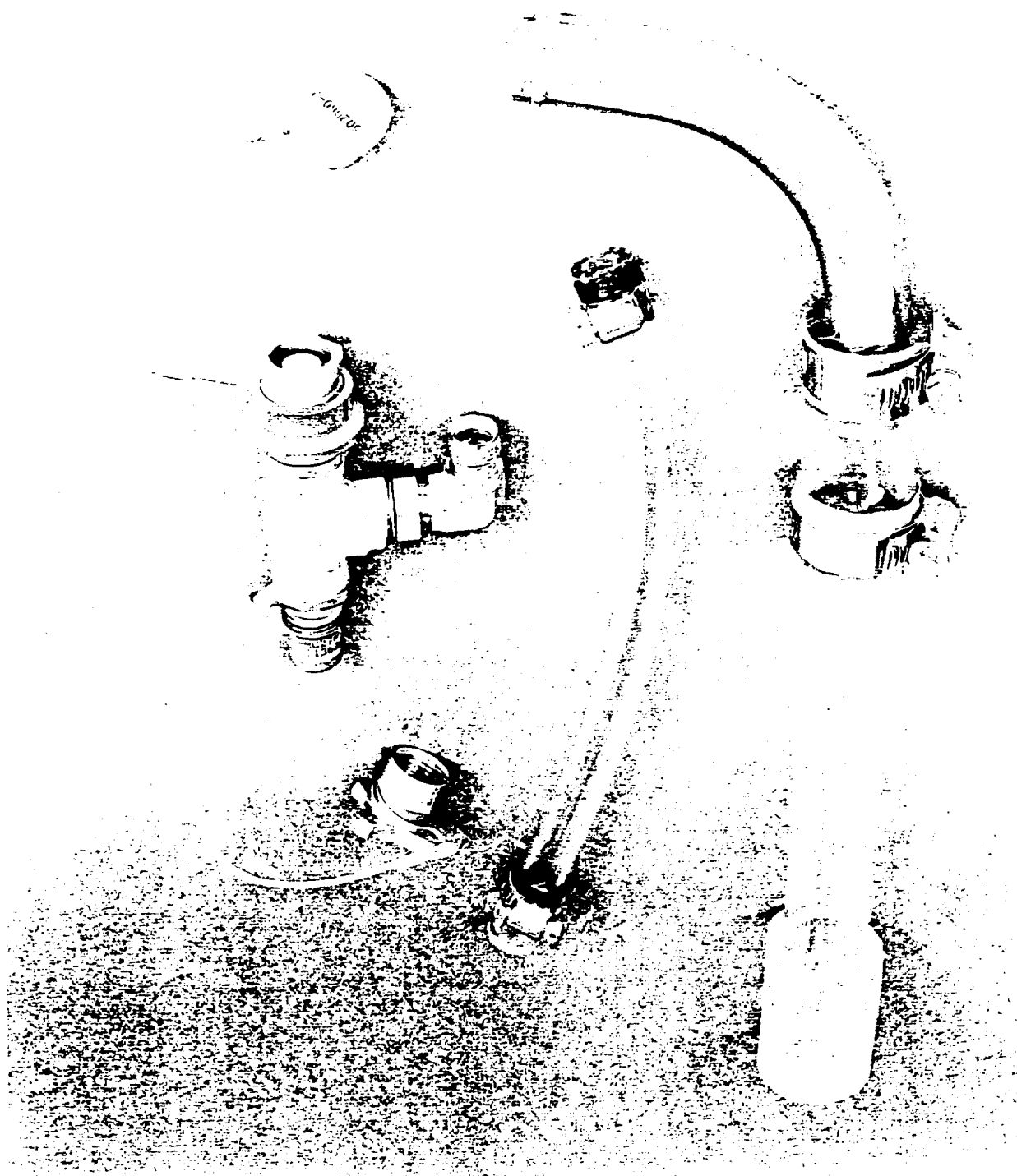


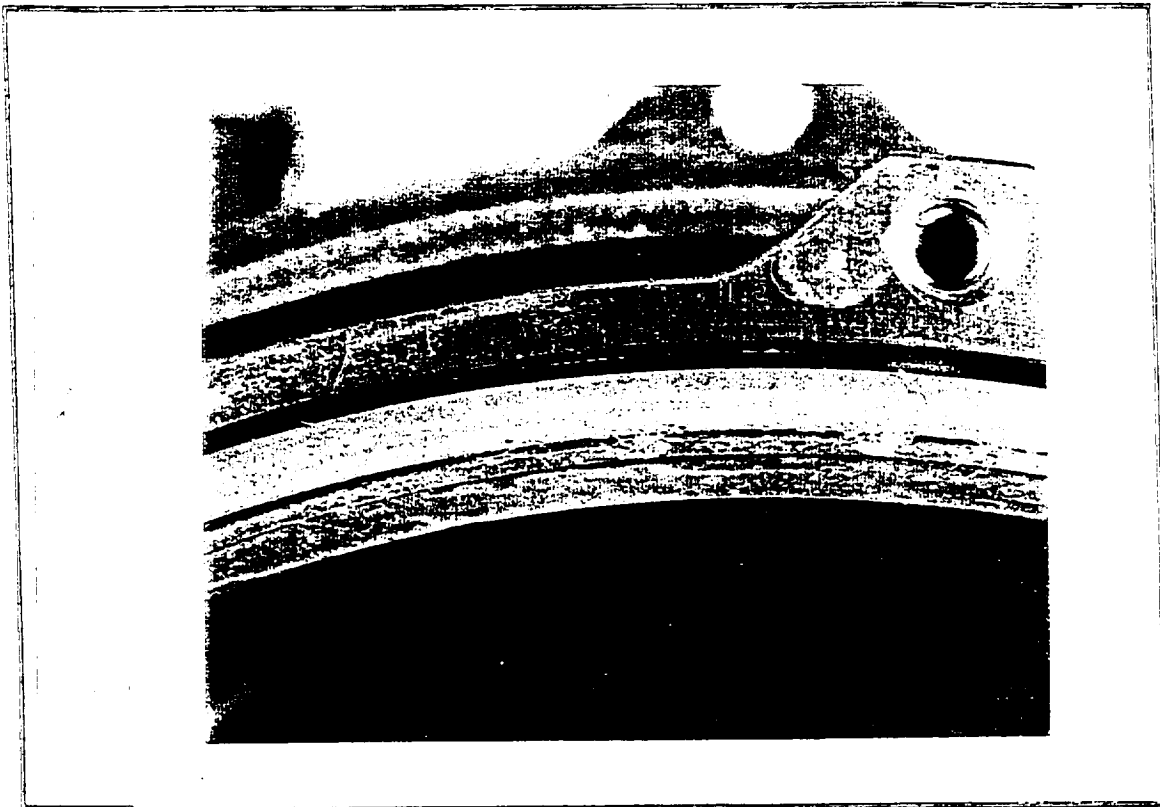
Figure 26

Post Test Teardown of the Check Valve Showing a
Clean Seat, Spring, and Poppet

Post Test Disassembly of the Portable Urine Collection
Rig Plumbing Showing No Evidence of Urine Deposits

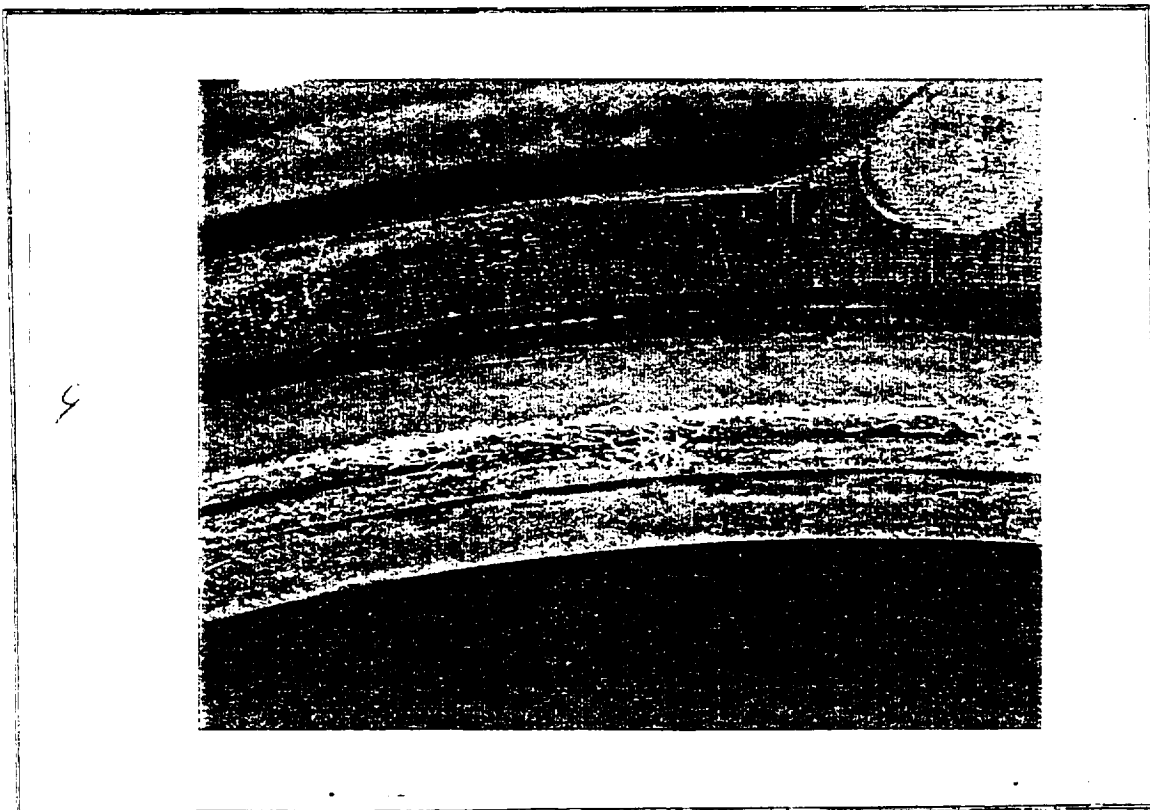
Figure 27





ID #4
 MAG. 5X
 LOCATION
 DRUM SEAL
 NOTES

 ETCHANT



ID #4
 MAG. 5X
 LOCATION
 DRUM SEAL
 NOTES

 ETCHANT

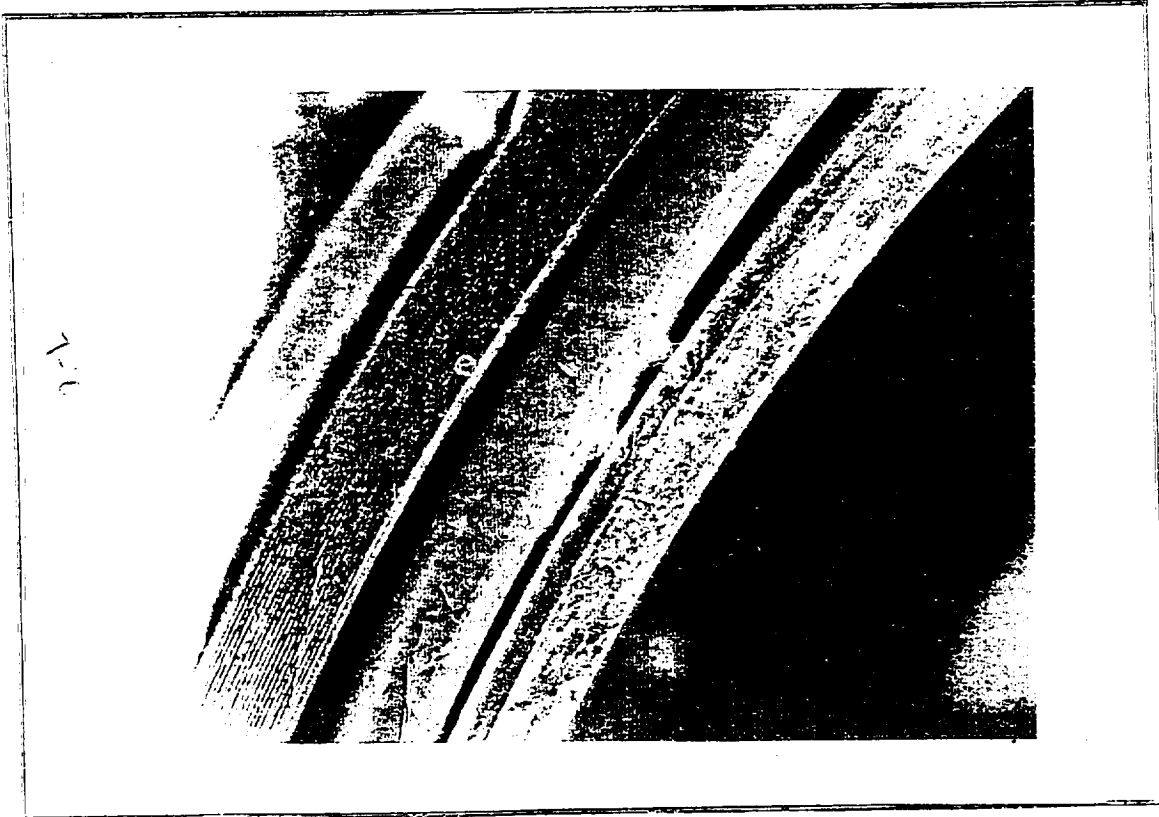
Figure 28

Post Test Examination of the Separator Drum Seal (I.D. #4)



ID 7-8
 MAG. 2X
 LOCATION
DRUM SEAL
 NOTES

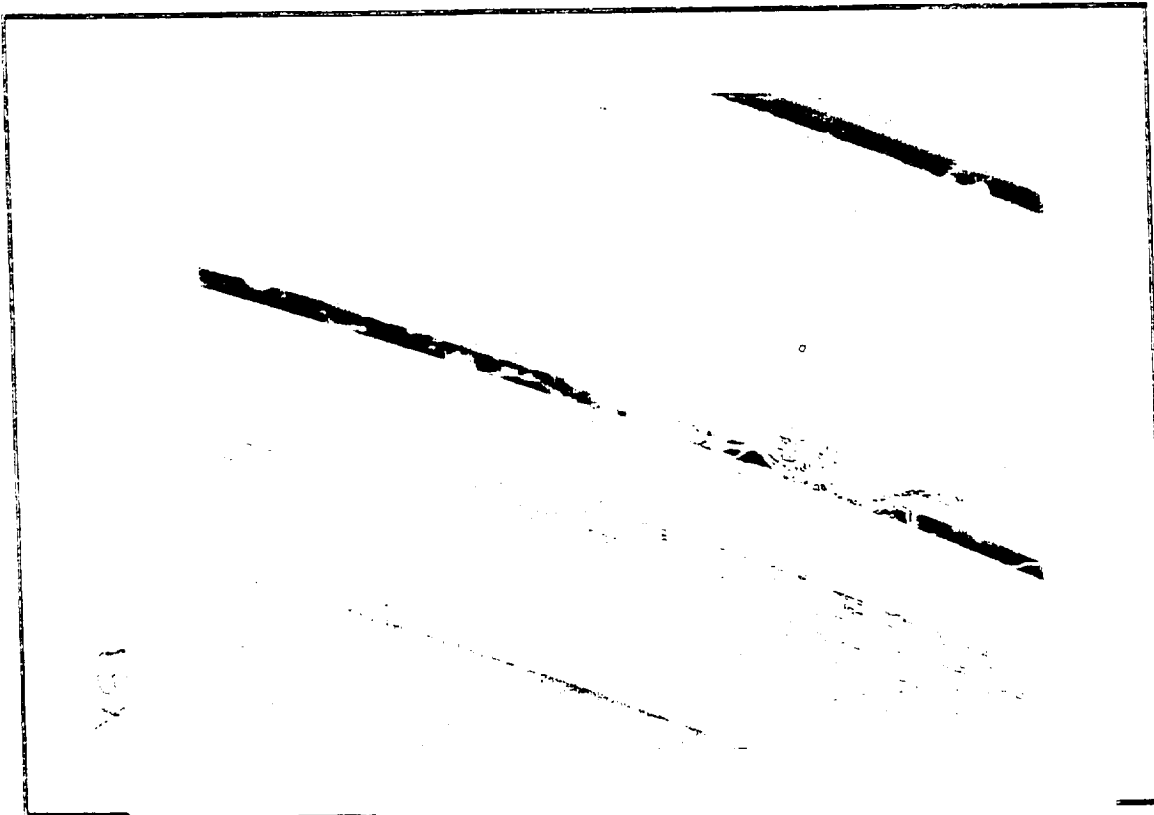
 ETCHANT



ID 7-8
 MAG. 5X
 LOCATION
DRUM SEAL
 NOTES

 ETCHANT

Figure 29 Post Test Examination of the Separator Drum Seal (I.D. #7-8)



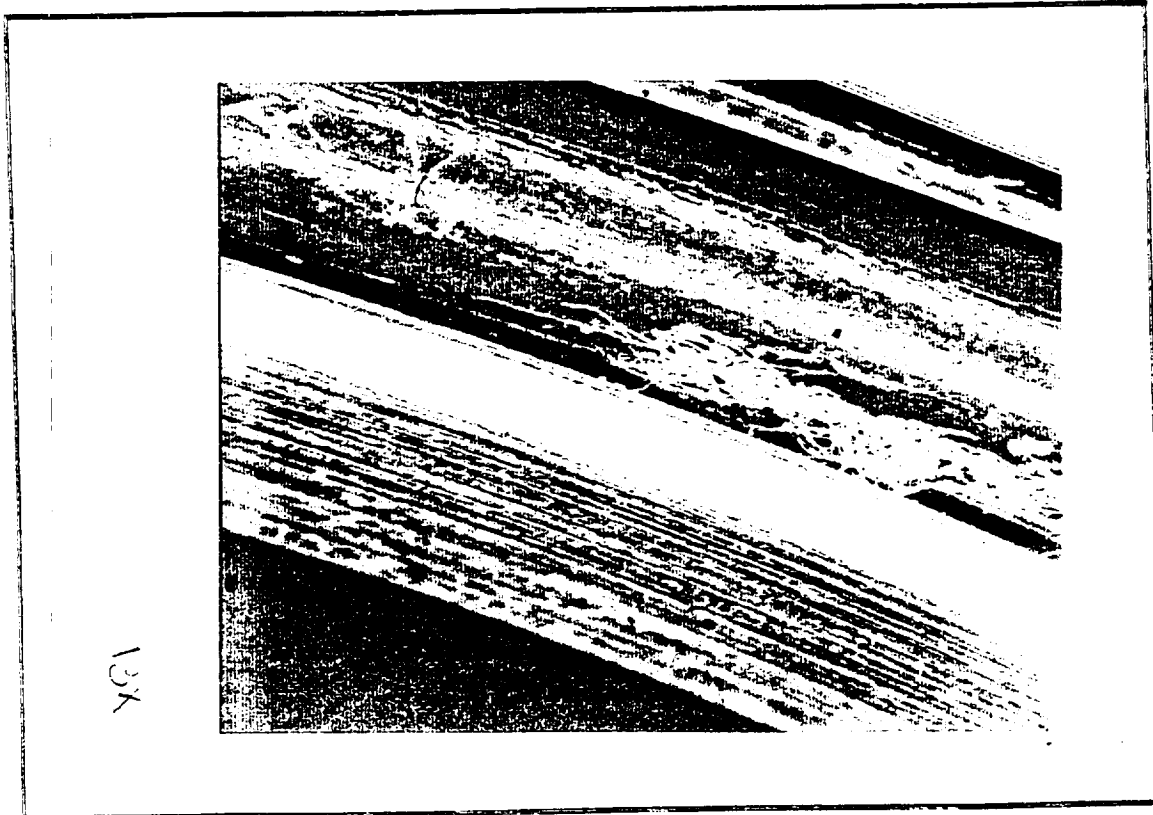
ID _____

MAG. 13X

LOCATION
DRUM SEAL

NOTES _____

ETCHANT _____



ID _____

MAG. 13X

LOCATION
DRUM SEAL

NOTES _____

High
mag
area

ETCHANT _____

Figure 30 Post Test Examination of the Separator Drum Seal (Mag. 13X)

Microbial Air Sampling of the Urine Separator with Pretreat - During the operation of the portable urine collection test rig in the men's bathroom, it was decided to conduct a microbial air sampling survey of the pretreat test. On 25 and 27 April 1995 several air samples were taken at three locations in the air side of the urine collection rig. Data indicated that air entering the portable urine collection test rig in the bathroom, which contained upwards of 192 CFU/M³ was reading less than the detectable level of 12 CFU/M³ at the outlet of the separator fan. The complete test and evaluation are presented in Appendix H.

Oxone/PEG Tablet Long Term Storage

A test was conducted to determine the long term storage stability of the Oxone/PEG tablets. Several tablets were processed on 12/6/94 and analyzed for the percentage active compound (KHSO₅) in the tablet. At this point in the program the tablets were processed with 10% PEG which would relate to the same percentage reduction from the maximum of 43% active component per the Oxone MSDS. The theoretical maximum active component of a 10% PEG tablet would be 39.7%. Table D shows the results for tablet storage with 10% PEG. The "bulk" analysis which takes a complete tablet which is ground up to a uniform mixture and then analyzed. The bulk analysis appeared to provide more consistent results than the "center" or "surface" analysis. Later in the test program during the actual urine collection test the PEG percentage was reduced and finally standardized at 3.5%. For this configuration a new group of tablets were set aside for a second long term storage test which started on 23 February 1995 and the results are also shown in Table D. The results show a high degree of stability over the storage period from 2/23/95 to 5/2/95.

Table D UPIS Oxone/PEG Pellet - Long Term Storage Study

Start Date = 12/6/94

Bulk = 26.1% KHSO₅
 Surface = 38.8% KHSO₅
 Center = 31.7% KHSO₅

First Sample = 1/19/95

Open Air Storage
 Bulk = 26.5% KHSO₅
 Surface = 38% KHSO₅
 Center = 26.8% KHSO₅

Plastic Bag Storage
 Bulk = 26.5% KHSO₅
 Surface = 34.3% KHSO₅
 Center = 27.0% KHSO₅

Glass Vial Storage
 Bulk = 24.5% KHSO₅
 Surface = 25.7% KHSO₅
 Center = 25.7% KHSO₅

Second Sample = 2/20/95

Open Air Storage
 Bulk = 26.5% KHSO₅
 Surface = 37.2% KHSO₅
 Center = 25.9% KHSO₅

Plastic Bag Storage
 Bulk = 26.3% KHSO₅
 Surface = 33.0% KHSO₅
 Center = 25.8% KHSO₅

Glass Vial Storage***
 Bulk = 25.5% KHSO₅
 Surface = 28.9% KHSO₅
 Center = 25.7% KHSO₅

Start Date = 02/23/95

Bulk = 33.2% KHSO₅

First Sample = 03/21/95

Open Air Storage
 Bulk = 33.8% KHSO₅

Plastic Bag Storage
 Bulk = 33.0% KHSO₅

Glass Vial Storage
 Bulk = 33.0% KHSO₅

Second Sample = 05/02/95

Open Air Storage
 Bulk = 32.0% KHSO₅

Plastic Bag Storage
 Bulk = 33.2% KHSO₅

Glass Vial Storage***
 Bulk = 33.1% KHSO₅

*** Significant amounts of moisture can be observed on the inside walls of the glass vials.

CONCLUSIONS

The prototype urine pretreat prefilter which contains several solid Oxone tablets provided a safe, convenient, simple and reliable method of incorporating the Oxone pretreat into a two-phase urine/air flow stream in a microgravity. The preferred concept of the trade study was developed into the prototype version with the proper quantity of Oxone in one pretreat/prefilter assembly for routine changeout by an astronaut. The long term urine collection test conducted with a flight type urine separator demonstrated the actual use of the urine pretreat prefilter. The final tablet formation and percent of PEG binder met the primary goal of controlling the primary goal of controlling the dissolution rate to maintain an average of just slightly higher than 5.0 grams of Oxone per liter of urine.

A secondary goal that was met was demonstrated by a post test teardown of the hardware and plumbing after processing a total of 342 liters of urine over a seven (7) week time period. A visual examination indicated the internal wetted surface areas were as clean as the initial assembly with no urine deposits. The Oxone pretreat alone has sufficient acidity and antimicrobial control to maintain proper hardware cleanliness upstream of the H_2SO_4 injection point. An additional Oxone concentration test conducted demonstrated that microbial control and urine precipitate control could be maintained with average levels of Oxone down to 3 - 3.5 grams per liter.

Another advantage of the Oxone as demonstrated by this program is that the high degree of antimicrobial control is also maintained in the entrainment air stream. Incoming air which had readable CFU/M³ values of 193 were reduced within the separator to less than the detection limit of 12 CFUs/M³ at the separator fan outlet. This would greatly reduce the bacteria load on the downstream odor/bacteria filter.

RECOMMENDATIONS

Based upon the successful completion of the first phase of the Urine Pretreat Injection System test program, the following recommendations are being made:

- A second continuation phase of the Urine Pretreat Injection Test Program should be conducted to include the effect of other pretreat chemical (H_2SO_4).
- Consider the possibility of using the developed Oxone Urine Pretreat Prefilter in the Space Station stage 10 testing at NASA/MSFC in lieu of the present liquid Oxone Injection System.
- Consider using the solid Oxone tablet approach in the Shuttle WCS to minimize urine deposits buildup and post flight maintenance.

Trade Study

Appendix A

Trade Study Objectives

- ◆ To review various methods of injecting OXONE® into the C/US urinal upstream of the separator.
- ◆ To select a method for prototype and test.

Selection Criteria

- ◆ Simplicity of Design
- ◆ Simplicity of Use
- ◆ Manufacturing Cost
- ◆ Handling Ease
- ◆ Safety
- ◆ Reliability
- ◆ Ease of Maintenance
- ◆ Maintenance Time
- ◆ System Envelope
- ◆ System Weight
- ◆ Logistics Supplies Envelope
- ◆ Logistics Supplies Weight
- ◆ Usability in ISSA C/US
- ◆ Material Compatibility
- ◆ Development Risk
- ◆ Development Cost
- ◆ Air Flow Impedance
- ◆ Power Consumption

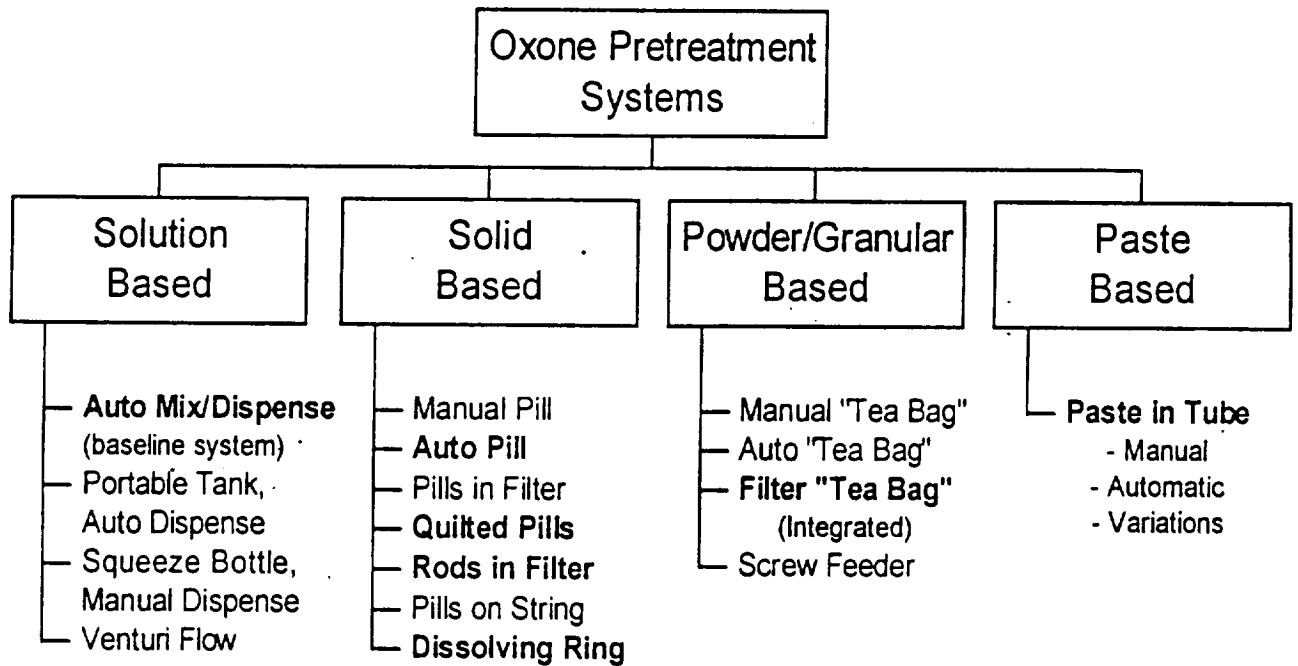
Concept Presentations & Comparisons

- ◆ Initial Concepts
- ◆ Selections for Further Evaluation
- ◆ Final Comparison

Initial Concepts...

- ◆ **Solution Based Systems** - introduce an Oxone/water solution into the urinal either automatically with pumps and injectors, or manually with a squeeze bottle.
- ◆ **Solid Based Systems** - introduce an Oxone/binder solid into the urinal in an active or passive manner. Solid designed to dissolve per micturation or over 24 hour period.
- ◆ **Powder/Granular Based Systems** - introduce an Oxone powder or grains to the urine stream in an active or passive manner. Per micturation or 24 hour doses possible.
- ◆ **Paste Based Systems** - introduce an Oxone paste to the urine stream, either automatically or manually, via a squeeze tube or syringe.

...Initial Concepts



Selections for Further Evaluation:

- ◆ Quilted Pills
- ◆ Pills in Filter
- ◆ “Tea Bag” Integrated Filter
- ◆ Dissolving Ring
- ◆ Paste System
- ◆ Auto Pill Injection
- ◆ Rods in Filter
- ◆ Automatic Solution Injection

Pills in Filter

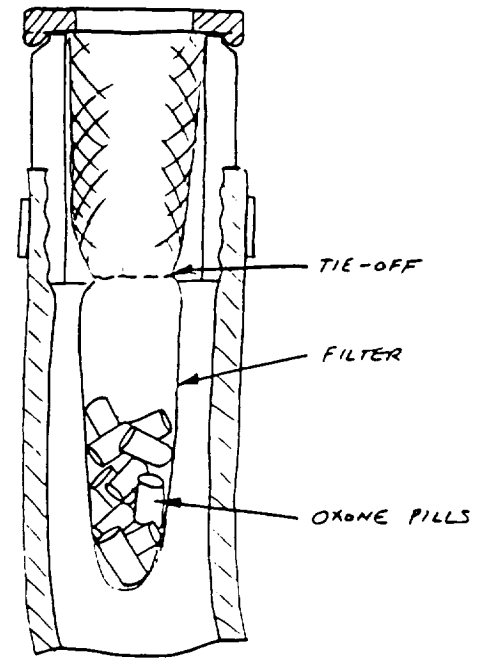
◆ Pros:

- simple design

◆ Cons:

- high ΔP .

- ◆ Conclusion - high bulk volume of pills required for 24 hr supply of Oxone will create excessive air flow obstruction.



Quilted Pills

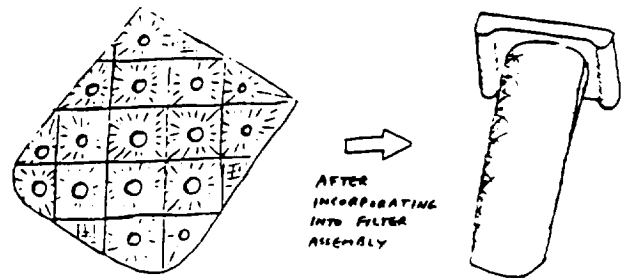
◆ Pros:

- may reduce pressure drop potential over pills-in-bag concept.

◆ Cons:

- requires long filter.
- complicated filter sock

- ◆ Conclusion - the number of pills required for 24 hr supply would make the filter prohibitively long.



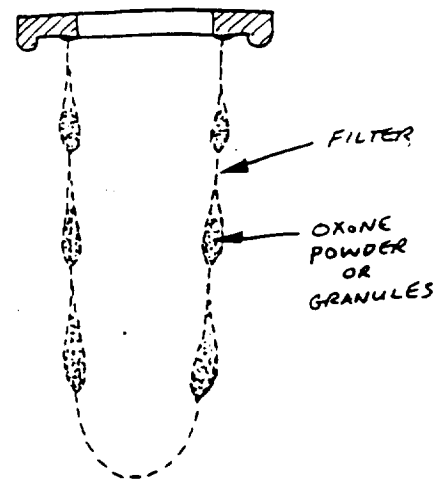
“Tea Bag” Integrated Filter

◆ Pros: ?

◆ Cons:

- dusting potential
- difficult to achieve dissolution over 24 hr period.
- high ΔP or long filter required for 24 hr design.

- ◆ Conclusion - this method has no outstanding features which would dictate its use.



Dissolving Rings

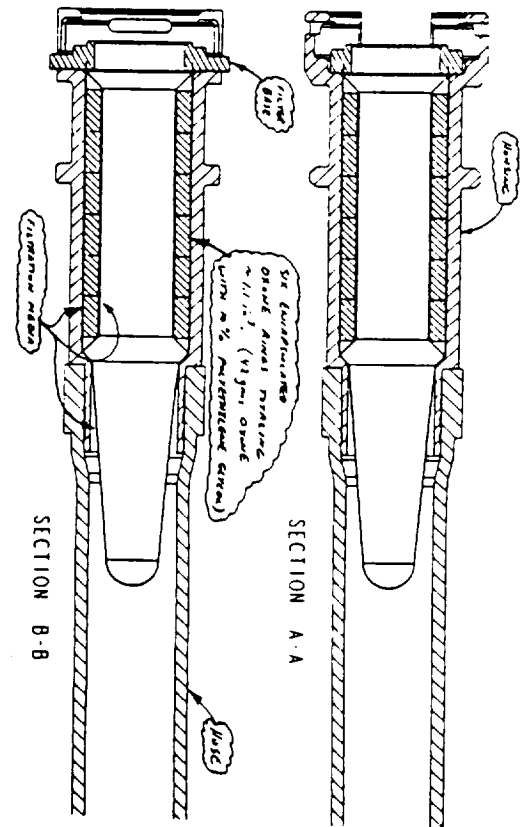
◆ Pros:

- reduces ΔP problems associated with other solids based concepts.

◆ Cons:

- increases urinal housing envelope.
- fragile form of solid, difficult to insert in urinal.

- ◆ Conclusion - negative aspects of this concept outweigh the ΔP benefit.



Paste System

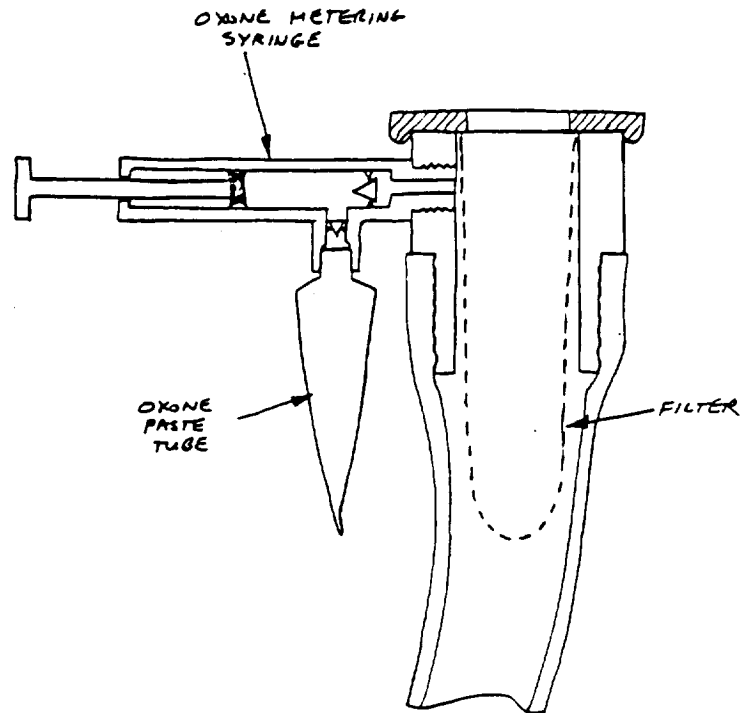
◆ Pros:

- per micturation dosage.

◆ Cons:

- requires paste development.
- back contamination prone.

- ◆ Conclusion - paste development risk and likelihood of contamination problems make this concept undesirable.



Auto Pill Injection

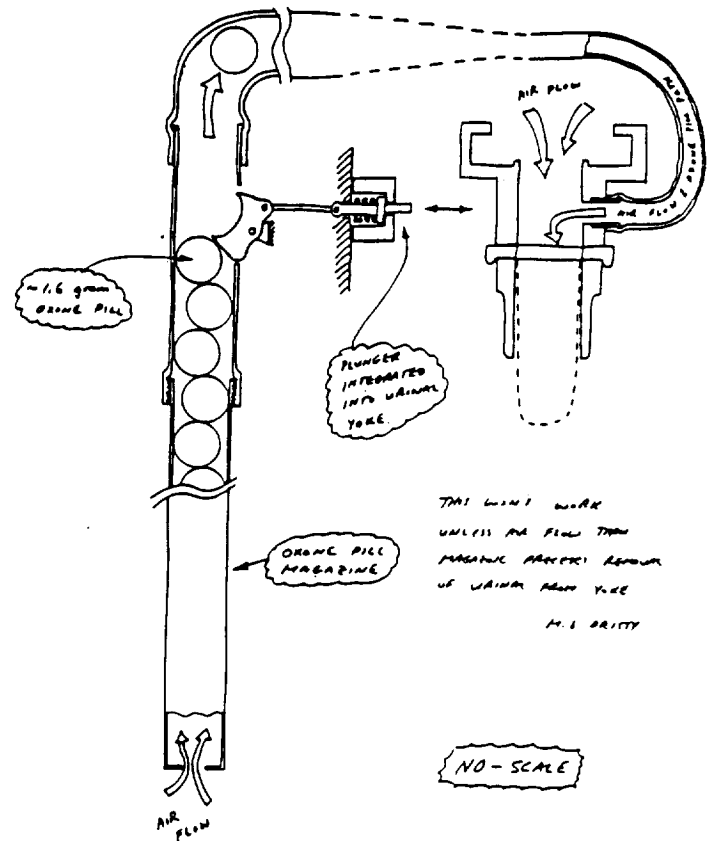
◆ Pros:

- per micturation dosage.

◆ Cons:

- relatively complex system.

- ◆ Conclusion - this concept doesn't offer any substantial benefits that would offset its cost & development risk.



Rods in Filter

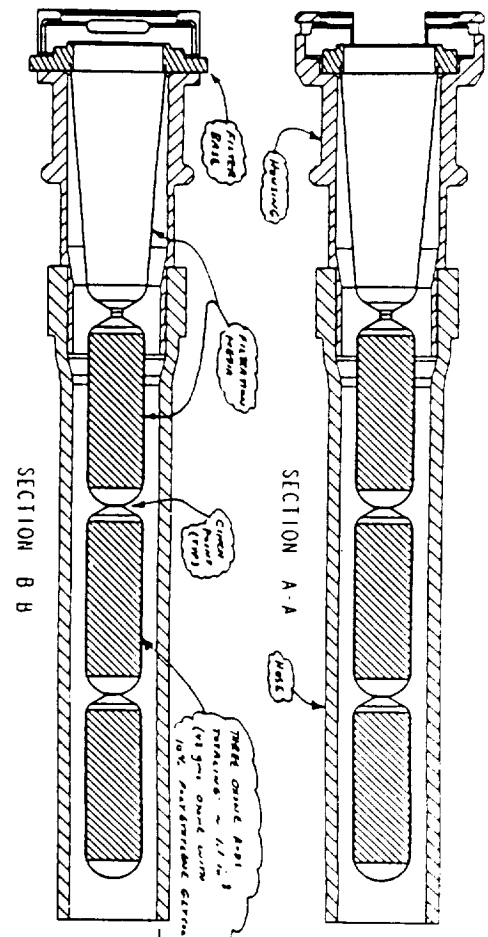
◆ Pros:

- simple design.
- less ΔP than other solid concepts.

◆ Cons:

- small ΔP , but acceptable.

- ◆ Conclusion - this concept offers benefits of simplicity in design and use which out-weigh the small flow impediment. This concepts should be compared to the baseline concept.



Auto Solution Injection

(Baseline Concept)

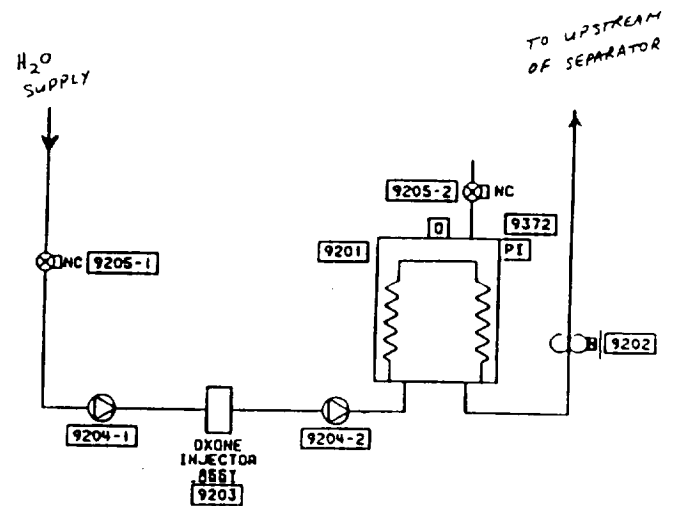
◆ Pros:

- per micturation dosage.
- no ΔP problems.

◆ Cons:

- complex system.
- substantial development risks.

- ◆ Conclusion - this system is complex and costly, but may be able to take advantage of Russian designs already developed.



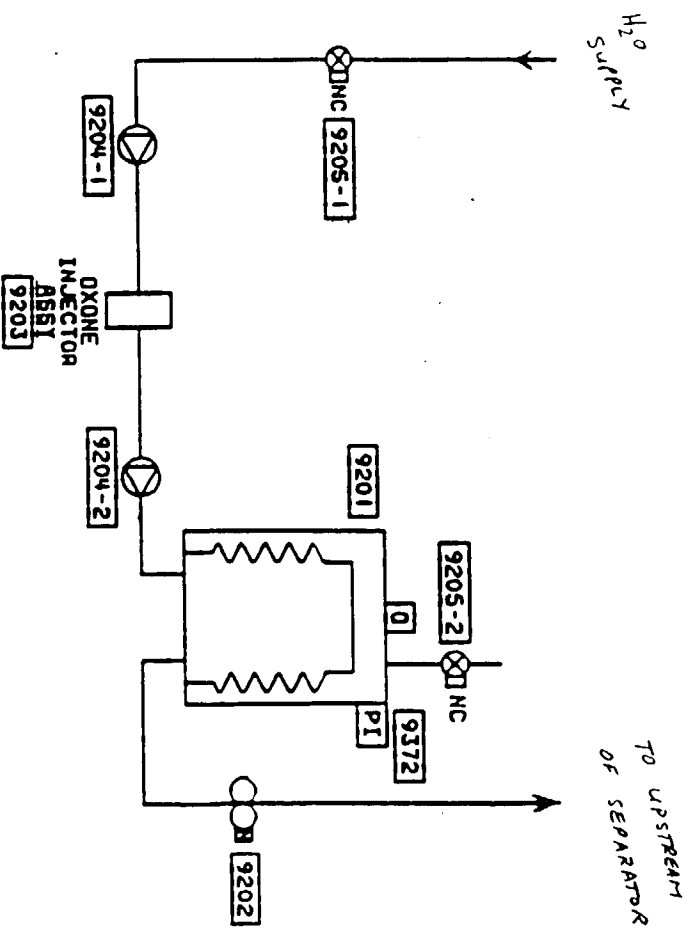
Final Comparison

Original Baseline System (Automatic
Solution Injection Systems)

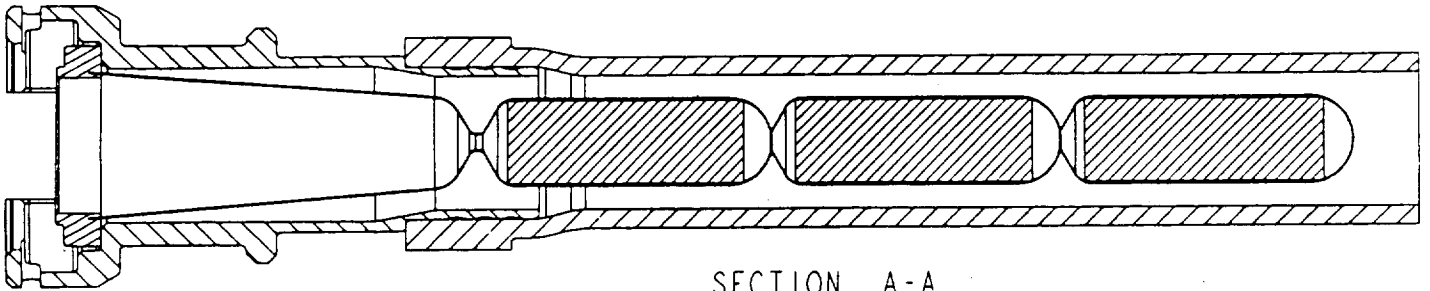
Vs.

Passive Solid Oxone System
(Rods in Filter)

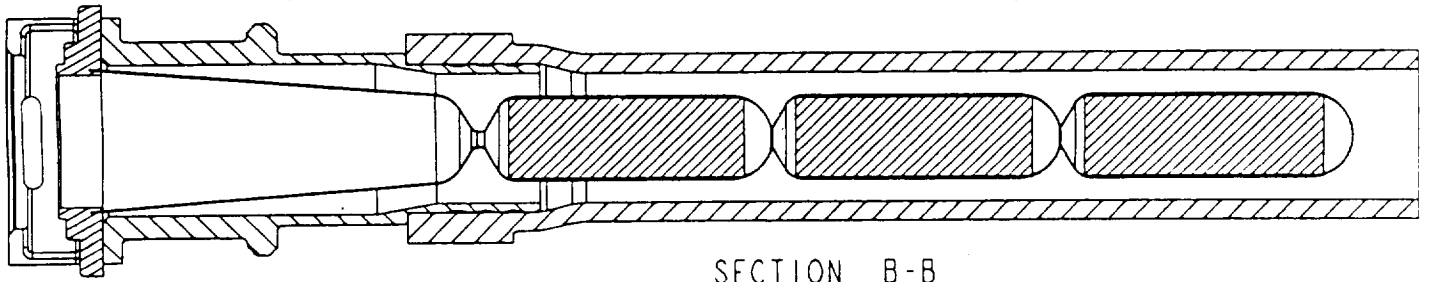
Automatic Solution Injection System (Typical)



Passive Solid System (Typical)



SECTION A-A
SCALE 1.000



SECTION B-B
SCALE 1.000

Comparison...

Auto Solution Injection Vs. Passive Solid

Criteria	Auto Soln. Inj.	Passive Solid
Simplicity of Design	Complex	Very Simple
Simplicity of Use	Simple	Simple
Manufacturing Cost	Expensive MORE	Very Inexpensive LESS
Handling Ease	Easy	Easy
Safety	Safe	Safe X
Reliability	Reliable	More Reliable
Ease of Maintenance	Difficult ORU REPLACEMENT	No Maintenance Req'd
Maintenance Time	Lengthy, but Infrequent	None
System Envelope	Large R	None
System Weight	Heavy ER	Essentially None

...Comparison

Auto Solution Injection Vs. Passive Solid

Criteria	Auto Soln. Inj.	Passive Solid
Logistic Supply Envelope	Larger SAME	Small SAME
Logistics Supply Weight	Heavier SAME	Light SAME
Usability in ISSA C/US	Usable	Usable
Material Compatibility	No Problems Expected	No Problems Expected
Development Risk	High	Low
Development Cost	High	Low
Air Flow Impedance	None	Higher, but Acceptable

Conclusions & Recommendation

- ◆ A 24 hour, filter-integrated solid Oxone pretreatment system has all the benefits of an automated solution injection system without the associated complexity, weight, envelope, high development cost & risk, and high manufacturing cost.
- ◆ HSSSI recommends the development and testing of the “Rods in Filter” type Oxone pretreatment system.

Prototype Hardware Drawings

Appendix B

FOR REVISIONS SEE SHEET 2

DWG NO SVSK116871 SH 5 DWG REV -

INDEX

REV	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29
SHEET	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	21	22	23	24	25	26	27	28	29

- DENOTES ORIGINAL ISSUE

ELEC DSGN																													
SYS DSGN																													
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TABLET, OXONE®

SIZE A CAGE CODE 73030 DRAWING NO. SVSK116871

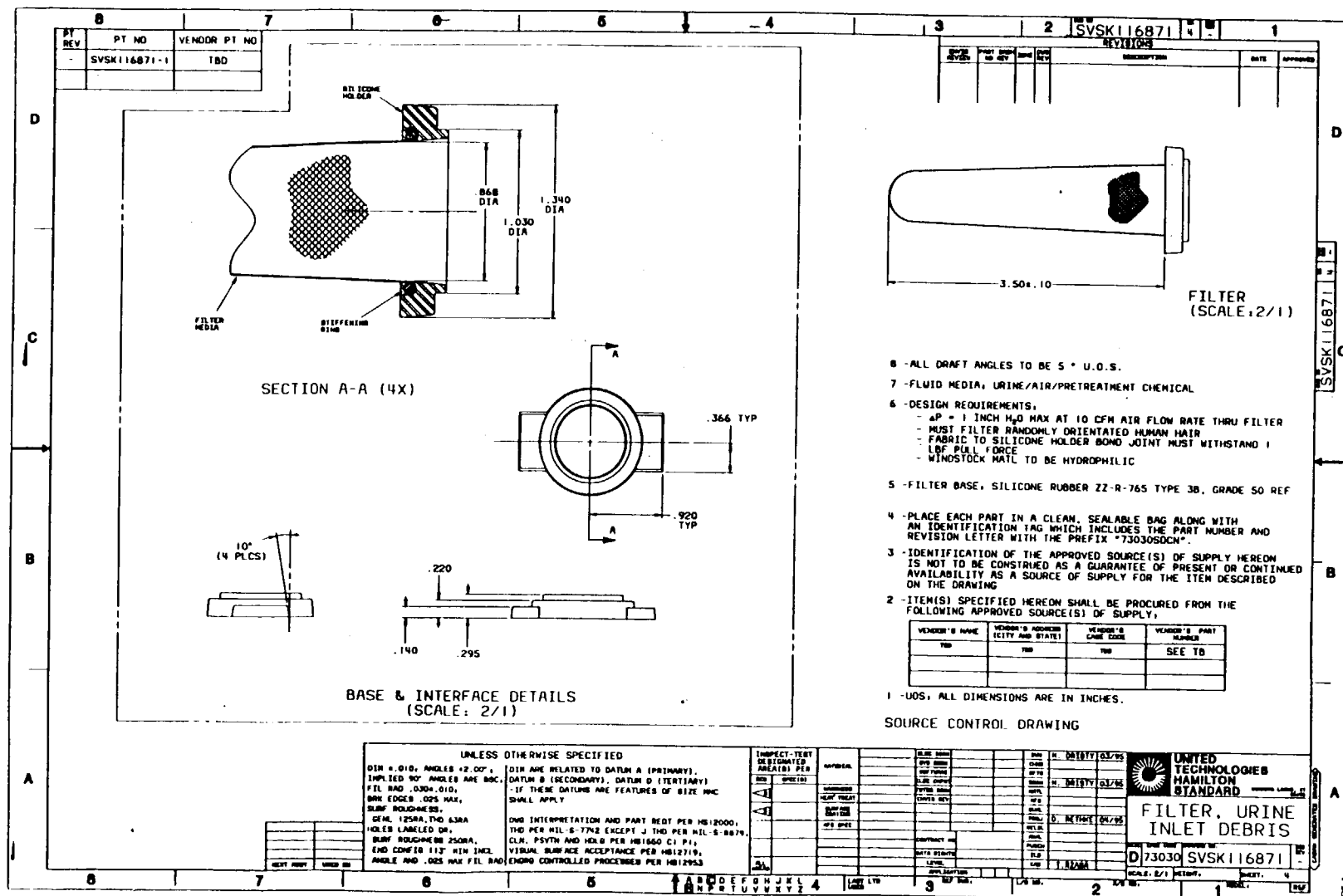
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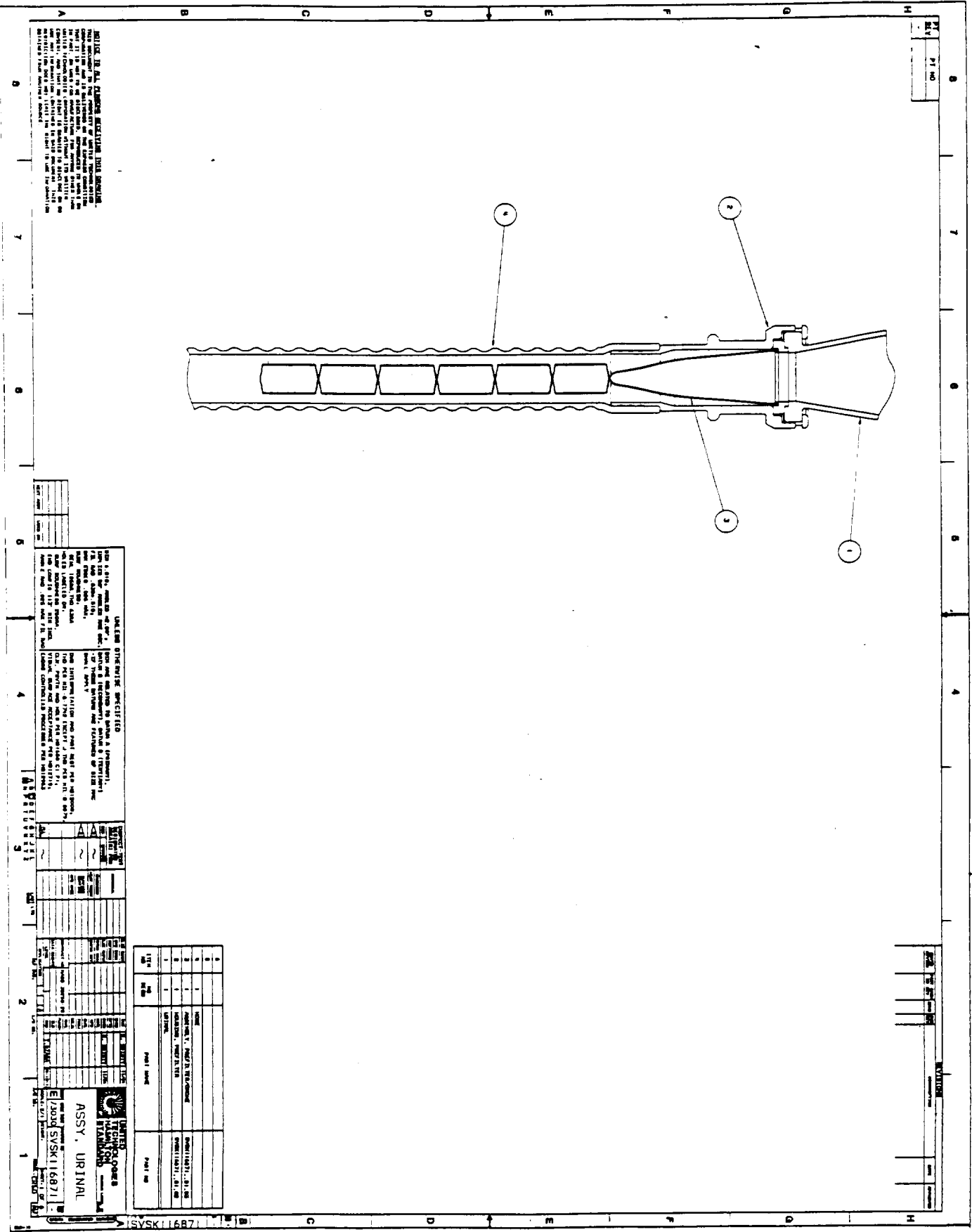
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REV	DATE	BY	CHKD	DESCRIPTION
1	10/11/67
2	10/11/67
3	10/11/67
4	10/11/67
5	10/11/67
6	10/11/67
7	10/11/67
8	10/11/67

UNITED STATES GOVERNMENT
ARPA
ASSY. URINAL
SVSK116871

Oxone
Product Specification
and
Data Sheet

Appendix C

DU PONT

PRODUCT SPECIFICATION

PRODUCT NAME: "OXONE" MONOPERSULFATE COMPOUND

Number: 3611

DATE ISSUED: 02/25/93
DATE SUPERSEDED: 05/22/89

SIMILAR NAMES: Potassium Peroxymonosulfate
Potassium Monopersulfate

Property	Units	Minimum	Maximum	Typical Analysis	Test Method
LES SPECIFICATIONS:					
Active Oxygen	(%)	4.5		4.7	07000.475.04.ME
Particle Size	(%)	100		100	07000.295.01.ME
Thru USS #20 Sieve					
Particle Size	(%)	95		100	07000.295.01.ME
Thru USS #30 Sieve					
Particle Size	(%)	5	35	20	07000.295.01.ME
Thru USS #100 Sieve					
Particle Size	(%)		10	5	07000.295.01.ME
Thru USS #200 Sieve					
Particle Size	(%)		5	2	07000.295.01.ME
Thru USS #325 Sieve					
Water	(%)		0.1	0.04	07000.570.01.ME

APPEARANCE: White, granular, free-flowing powder.

OTHER INFORMATION: Strong, odorless oxidant.

PHYSICAL DATA:

Property	Typical Value
Molecular Weight	614.7
Potassium Monopersulfate (KHSO5) (%)	44.7
Bulk Density,	
lb/ft ³	72-79
g/cm ³ (Mg/m ³)	1.15-1.27
pH @ 25C (77F)	
1% solution	2.3
3% solution	2.0
Solubility g/100g H ₂ O, 20C (68F)	25.6

For Further Information, Contact:

DuPont Company
Du Pont Chemicals
Wilmington DE. 19898
800 441-9408

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DuPont Specialty Chemicals

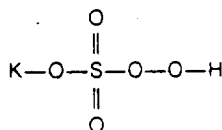
RETHKE

DATA SHEET

OXONE® Monopersulfate Compound

OXONE® Monopersulfate Compound is a white, granular, free-flowing powder ideal for applications requiring a strong, odorless oxidant. This peroxygen compound is particularly useful in formulated specialty products such as denture cleaners, swimming pool products, laundry bleaches, scouring powders, and bowl cleaners. OXONE is also used as a selective oxidant in the manufacture of organic chemicals and as a processing aid in repulping internal broke or a secondary fibers furnish.

OXONE is a triple salt with the formula $2\text{KHSO}_5 \cdot \text{KHSO}_4 \cdot \text{K}_2\text{SO}_4$. The active component, potassium monopersulfate, has the chemical structure:



The physical properties and typical analyses of OXONE are shown in Table I.

Solubility

OXONE is very soluble in water as shown in Table II. This excellent solubility offers a distinct advantage over less soluble dry oxidants, such as sodium perborate, particularly at relatively low temperatures. At 20°C, the solubility of OXONE in water is greater than 25 weight percent.

Water-ethanol, water-acetic acid, and water-ethanol-acetic acid mixtures are good solvent combinations when solvents other than 100% water are desired.

TABLE I
DU PONT OXONE® PHYSICAL PROPERTIES
AND TYPICAL ANALYSES*

Molecular Weight	614.7
Active Oxygen, % min.	4.5
% average analysis	4.7
% theoretical (triple salt)	5.2
Active Component (KHSO ₅), % min.	42.8
Bulk Density, g/cm ³ (Mg/m ³)	1.12-1.20
lb/ft ³	70-75
Particle Size through USS #20 Sieve, %	100
through USS #200 Sieve, % max.	10
(also see Table III)	
pH	
25°C (77°F)	
1% solution	2.3
3% solution	2.0
Solubility g/100 g H ₂ O, 20°C (68°F)	25.6
(also see Table II)	
Moisture Content, %	0.1
Stability, % active oxygen loss/month	<1
Standard Electrode Potential (E°), volts	-1.44
Heat of Decomposition, kJ/kg	251
Btu/lb	108
Thermal Conductivity, W/m-K	0.161
Btu-in/hr-ft ² -°F	0.093

* This table gives typical properties based on historical production performance. Du Pont does not make any express or implied warranty that this product will continue to have these typical properties.

* Reg. U.S. Pat. and Tm. Off., Du Pont Company. OXONE® Monopersulfate Compound is made only by Du Pont.

NOTICE: OXONE® MONOPERSULFATE COMPOUND CAUSES IRRITATION.
See Personal Safety and First Aid on page 3.

The information set forth herein is furnished free of charge and is based on technical data that Du Pont believes to be reliable. It is intended for use by persons having technical skill and at their own discretion and risk. Since conditions of use are outside our control, we make no warranties, express or implied, and assume no liability in connection with any use of this information. Nothing herein is to be taken as a license to operate under or a recommendation to infringe any patents.

TABLE II SOLUBILITY OF OXONE® MONOPERSULFATE COMPOUND IN WATER		
Temperature		g OXONE/100 g H ₂ O
°C	°F	
20	68	25.6
27	80	26.8
49	120	30.0
60	140	31.5
71	160	33.5

TABLE III TYPICAL PARTICLE SIZE ANALYSIS OF DU PONT OXONE® MONOPERSULFATE COMPOUND	
U.S. Screen Size (Mesh)	Approx. Weight % On Screen, Cumulative
30	1
70	68
100	84
200	98
325	100

Stability

Temperature and pH can affect the stability of solutions of OXONE® Monopersulfate Compound (Figures 1 and 2).

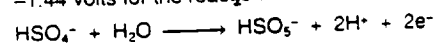
At pH values below 6 and above 12, solutions of OXONE are relatively stable; however, at pH 9 a point of minimum stability exists. At pH 7.5 or lower, active oxygen exists as HSO₅⁻, while at pH 12 the active oxygen species is SO₅²⁻. Between pH 7.5 and 12 both ionic species exist in solution and minimum stability corresponds to equal concentrations of HSO₅⁻ and SO₅²⁻.

OXONE solutions are not as sensitive to trace metal impurities as most peroxygen compounds. However, cobalt, nickel, copper, and manganese ions do catalyze the decomposition of OXONE with the evolution of oxygen gas.

CHEMICAL PROPERTIES

Oxidation Potential

The standard electrode potential (E°) of OXONE is -1.44 volts for the reaction:



This high potential suggests many room temperature oxidations with OXONE: halide ion to halogen, ferrous ion to ferric, manganous ion to manganic, and hydrogen peroxide to oxygen.

Formulation

OXONE is compatible with anhydrous salts such as sodium sulfate, sodium tripolyphosphate, tetrasodium pyrophosphate, sodium carbonate, and sodium metasilicate. Conventional surfactants such as alkyl aryl sulfonates and limited quantities of nonionic detergents may also be used in formulations of OXONE.

FIGURE 1

STABILITY OF A 1% SOLUTION OF
OXONE® AT VARIOUS TEMPERATURES

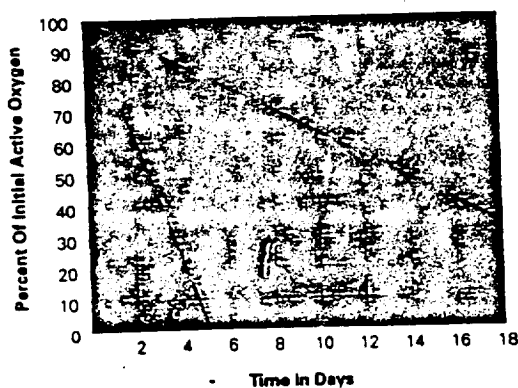
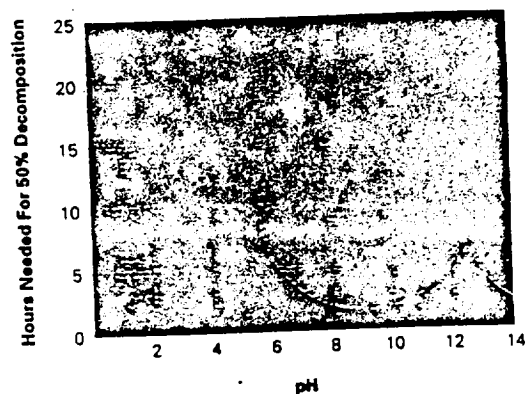


FIGURE 2

EFFECT OF pH* ON STABILITY OF A
3% SOLUTION OF OXONE® AT 32°C (89.6°F)



* pH adjusted with KOH.

CHEMICAL REACTIONS

OXONE[®] Monopersulfate Compound reacts with many organic compounds in aqueous or solvent-water solutions to convert:

1. Phenol to quinone (Elbs persulfate oxidation).
2. Cyclic ketones to lactones.
3. Toluene to benzoic acid.
4. Diphenylmethane to benzophenone.

OXONE can convert:

1. Olefins to glycols or glycol esters, depending upon the solvent system selected.
2. Cyclohexene to trans- rather than cis-cyclohexanediol.
3. Primary aryl amines to nitroso compounds.
4. Pyridine to pyridine-N-oxide by a slurry of **OXONE** in glacial acetic acid.

Epoxides have not been isolated from reaction systems of **OXONE** and olefins.

OXONE can initiate the free radical polymerization of typical vinyl monomers such as vinyl acetate, ethyl acrylate, and acrylonitrile.

An atypical reaction is the conversion of toluene to benzyl halide by heating with a dry mixture of **OXONE** and sodium chloride or sodium bromide.

Detailed information on the use of **OXONE** in organic reactions appears in "Oxidation of Organic Substances by Potassium Peroxymonosulfate" by R. J. Kennedy and A. M. Stock, *J. Org. Chem.*, 25, 1901 (1960).

PERSONAL SAFETY AND FIRST AID

Health Hazards

OXONE Monopersulfate Compound has a low order of toxicity when taken internally. The approximate lethal dose (ALD) for rats is 2250 mg/kg.

OXONE is irritating to the eyes, skin, nose, and throat due to its acidity and oxidizing properties, and may cause allergic reactions in sensitive individuals. Du Pont observes an airborne exposure limit to **OXONE** dust of 1 mg/m³, 8-hour time weighted average.

Safety Precautions

Persons handling **OXONE** should avoid contact with skin, eyes, or clothing. Avoid breathing dust. Wash thoroughly after handling and launder contaminated clothing before reuse. Exposure can be minimized by providing adequate ventilation and by wearing rubber- or plastic-coated gloves and chemical safety goggles when handling **OXONE**.

Site Facilities

The following safety equipment should be easily accessible in all areas where **OXONE** is handled or stored:

Safety Showers with quick opening valves that stay open. Water should be supplied through insulated lines.

Water Hydrant and Hose or other means of flushing spills with large volumes of water under low pressure.

Eye Wash Fountains or other means for washing the eyes with a gentle flow of tap water.

First Aid

In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Flush skin with water. If inhaled, remove to fresh air. Call a physician.

APPLICATIONS

The high oxidation potential of **OXONE** suggests its application in various cleaning compounds where it is desired to eliminate oxidizable discolorations. **OXONE** can be blended with a wide variety of conventional additives including sodium perborate monohydrate, diethylenetriamine pentaacetic acid (DTPA), tetrasodium pyrophosphate, sodium tripolyphosphate, sodium metasilicate, sodium carbonate, sodium bicarbonate, sodium sesquicarbonate, citric or tartaric acid, wetting agent or detergent, sodium sulfate, and fragrance. Formulations can be sold as free-flowing powders or can be tableted.

Since **OXONE** is acidic, it is usually buffered to near neutral or alkaline pH when compounded into cleansers.

Formulations of **OXONE** containing chloride ions can be used to generate low concentrations of active chlorine in cleaning systems. The active chlorine can be present as hypochlorous acid or hypochlorite ion, depending on the system pH; it is necessary to use sufficient alkali in the formulation to avoid generation of elemental chlorine, which is a highly toxic gas. **OXONE** that is not converted to active chlorine will continue to function as active oxygen.

The above additives must be anhydrous; otherwise, undesirable reactions may occur between the acidic solution of **OXONE** and the additives.

Denture Cleaners

The properties of **OXONE** have been shown to be particularly adaptable to denture cleaner formulations because of its ability to decolorize food and other organic stains. To obtain the desired pH and cleansing properties, a general purpose denture cleaner formulation should contain at least 25% **OXONE**, and any of the additives listed above.

Industrial/Institutional Laundry Bleach

OXONE[®] Monopersulfate Compound can be readily formulated with conventional anhydrous alkaline fillers into a stable, free-flowing, dry bleach. In formulating a dry bleach with **OXONE**, sufficient alkali must be used to ensure a pH of 9–10 in the laundry use. A suggested mixture of 35% **OXONE** and 65% light granular soda ash is simple, low cost, with low bulk density, and has an attractive appearance. In use, a concentration of at least 25 ppm of active oxygen is preferable in the laundry solution (approximately 4 ounces of the 35/65 mixture per 18 gallons of water will yield 25 ppm active oxygen). For stain removal, concentrations as high as 200 ppm active oxygen in hot water may be used. **OXONE** must be dissolved prior to contacting fabrics as it may cause dye damage under some conditions.

Bowl Cleaners

Toilet bowl cleaners for home use are composed primarily of sodium bisulfate with small amounts of detergent, fragrance, and corrosion inhibitor. The solution pH of these cleaners in use is 1–2. Functions of the acidic bowl cleaners are soil and stain removal. These product characteristics may be enhanced by including 1–3% **OXONE** in the bowl cleaner formulation.

SWIMMING POOL/SPA PRODUCTS

Swimming Pool/Spa Oxidizer ("Shocking Agent")

OXONE Monopersulfate Compound can be used as an auxiliary oxidant (shocking agent) in swimming pools and spas for the purpose of reducing the organic content of the water. The treatment, which is generally recommended at two-week intervals or whenever cloudiness is present, increases the clarity of the water and reduces eye burn and chlorine odor by destroying chloramines.

The excellent solubility of **OXONE** makes it ideal for addition to pools and spas by broadcast or via the filter basket.

Unlike chlorine-based shocking agents that super-chlorinate the pool, **OXONE** does not increase the chlorine level. Therefore the pool need not be closed except for a short period to fully circulate the **OXONE**.

In this application, **OXONE** is not a disinfectant and must be used in addition to an EPA (FIFRA) registered disinfectant.

OXONE/Sodium Bromide Disinfection

OXONE can also be used as one part of a two-part disinfectant system for spas and hot tubs with sodium bromide. **OXONE** oxidizes bromide to bromine, which is present as the active disinfectant HOBr in the pH range usually found in spas and hot tubs. This two-part disinfectant system has been EPA (FIFRA) registered by several

manufacturers of swimming pool chemicals; such registration is necessary before offering disinfectants for sale in the United States. Other jurisdictions may also require additional registrations.

Since the organic loading of a spa or hot tub is often greater than that of a swimming pool, chlorine-based disinfectants have a tendency to cause cloudiness and chlorine odor because of the formation of stable chloramines. Bromine-based disinfectants form bromamines, which are unstable and are recognized as good disinfectants. Bromine-disinfected spas have lower odor, less cloudiness, and cause less eye irritation than chlorine-disinfected spas.

For this use, it may be desirable to neutralize the acidity of **OXONE** by formulation with an anhydrous alkaline salt such as sodium carbonate. A mixture of 80% **OXONE**/20% sodium carbonate by weight gives a neutral pH. Dilution to control dosage may be done with anhydrous sodium sulfate.

PULP AND PAPER INDUSTRY

OXONE Monopersulfate Compound can be used as a processing aid in repulping internal broke or a secondary fibers furnish. Paper products containing polyamide epichlorohydrin wet strength resins can be effectively repulped to make the same grade paper. **OXONE** has been demonstrated on a variety of sanitary, food, packaging, and specialty papers containing wet strength resin and made from different furnishes. In addition, because of its dye destruction capability, **OXONE** can also be used to repulp certain colored papers.

Unlike chlorine-based repulping agents, **OXONE** does not produce AOX or other chlorinated organics. It also does not degrade the fiber or darken mechanical pulp. It is easier to handle and does not require any additional storage or delivery equipment. Under optimum conditions, **OXONE** reacts rapidly, thereby reducing the repulping time and increasing the productivity of the pulper. It is effective under commonly found pulping conditions.

TEST METHODS

Active Oxygen/Active Component

The active oxygen in **OXONE** Monopersulfate Compound or in mixtures of **OXONE** with other materials can be determined by using the standard iodometric titration. Iodine released from an acidic potassium iodide solution by the active oxygen in **OXONE** is titrated with standard sodium thiosulfate to a colorless end point. The following method is suggested.

1. Take four samples, one from each quarter of the material to be analyzed.
2. Blend samples by using a small blender or rolling in a container for about 5 minutes.
3. Empty the blended sample onto a glass pie plate or flat dish and take small samples at random to obtain 0.5 g for analysis.
4. Weigh the sample to the nearest 0.001 g.
5. In a 250 mL beaker equipped with stirrer add 50 mL distilled water, 5 mL of 20% sulfuric acid, and 10 mL 20% potassium iodide solution. Then add the weighed sample, and stir to completely dissolve it.
6. Titrate with 0.1 N sodium thiosulfate solution to a colorless end point that persists for 30 seconds. (Starch indicator should be used for enhancement of the end point.)
7. Calculate:

% active oxygen =

$$\frac{\text{mL thiosulfate} \times N \times 0.008 \times 100}{\text{sample weight (grams)}}$$

where *N* is the normality of the sodium thiosulfate.

$$\% \text{ active component (KHSO}_5\text{)} = \frac{\% \text{ active oxygen}}{0.1053}$$

Moisture Content

1. Using the sampling procedure above, weigh 10 g (± 0.01 g) in a tared aluminum dish.
2. Dry for 30 minutes in a $65^\circ\text{C} \pm 0.5^\circ\text{C}$ oven.
3. Cool in a desiccator and weigh.
4. Calculate:

$$\% \text{ moisture} = \frac{\text{original wt.} - \text{dry wt.}}{\text{original wt.}} \times 100$$

SHIPPING CONTAINERS

A moisture barrier package is recommended for formulated mixtures of OXONE. Du Pont ships OXONE in multiwalled, moisture-resistant, 25 kg (55.1 lb) net paper bags and in lined, 907 kg (2000 lb) bulk bags. OXONE Monopersulfate Compound is not regulated as a hazardous material by the Department of Transportation as of December 1987.*

* Due to changing government regulations such as those of the Department of Transportation, Department of Labor, U.S. Environmental Protection Agency, and the Food and Drug Administration, references herein may be superseded. The user should consult and follow the current governmental regulations, such as Hazard Classification, Labeling, Food Use Clearances, Worker Exposure Limitations, and Waste Disposal Procedures for the product described in this literature.

STORAGE AND HANDLING

Precautions in Use

OXONE® Monopersulfate Compound is a moderately strong oxidizer. It should be stored in a cool, dry location, away from combustible materials.

The mixture of OXONE with compounds containing halides or active halogens can cause release of the respective halogen if moisture is present. For example, mixture with sodium dichloroisocyanurate or with sodium chloride can cause release of chlorine gas; mixture with cyanides can cause release of hydrogen cyanide gas; and heavy metal salts such as those of cobalt, nickel, copper, or manganese cause the evolution of oxygen.

Like all other peroxygen compounds, OXONE undergoes very slow decomposition in storage, which also liberates heat. In order to provide sufficient surface area to dissipate the small amount of heat generated, OXONE should not be stored or processed in large masses exceeding a cube 4 ft on each side (64 ft³) or approximately 4500 lb OXONE. Storage of quantities of OXONE in excess of this limit can lead to runaway decomposition with liberation of large amounts of heat and oxygen gas.

If the internal temperature of the material exceeds 300°C (m.p. K_2SO_4), the material may fuse and could generate SO_2 or SO_3 gases.

Pallets of OXONE (less than 64 ft³/pallet) can be stacked if there are 2–3 inches of air space between pallets.

Grinding or intensive mixing may generate sufficient heat to fuse OXONE and cause the ignition of oxidizable material if present.

Spills

Spills and sweepings should be removed and the area thoroughly washed with water.

Dry Stability

When stored in a cool, dry place, OXONE is an exceptionally stable peroxygen compound. The rate of decomposition at these storage conditions should be less than 1% per month of contained active oxygen. Any mixture should be tested prior to packaging.

Materials of Construction

The primary consideration in choosing equipment for handling OXONE and solutions of OXONE is to prevent contamination of the product with rust or other catalytic metals. Thus, stainless steel, porcelain, glass, and many plastics may be considered suitable. Containers and small packages for OXONE should be of moisture barrier construction.

The use of OXONE and sodium chloride in formulations may cause mild corrosion problems, but the use of sodium nitrate or other chloride corrosion inhibitors will minimize this problem.

**Du Pont Chemicals
Wilmington, Delaware 19898**

U.S. Sales and Services

For placing orders or requesting additional product information,
please use our convenient 24-hour toll-free telephone number.
If you prefer, you can write to us.

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Oxone

Material Safety Data Sheet

Appendix D

RETHITE



Du Pont Chemicals

3661CR



Revised 10-JUN-1993

Printed 5-APR-1994

"OXONE" MONOPERSULFATE COMPOUND

CHEMICAL PRODUCT/COMPANY IDENTIFICATION

Material Identification

"OXONE" is a registered trademark of DuPont.

Corporate MSDS Number DU005614

CAS Number 70693-62-8

CAS Name POTASSIUM HYDROGEN PEROXYMONOSULFATE
SULFATE

Grade TECHNICAL

Tradenames and Synonyms

POTASSIUM PEROXYMONOSULFATE

Company Identification

MANUFACTURER/DISTRIBUTOR

DuPont
1007 Market Street
Wilmington, DE 19898

PHONE NUMBERS

Product Information 1-800-441-9442

Transport Emergency CHEMTREC: 1-800-424-9300

Medical Emergency 1-800-441-3637

COMPOSITION/INFORMATION ON INGREDIENTS

Components Material	CAS Number	%
POTASSIUM PEROXYMONOSULFATE	10058-23-8	43
POTASSIUM BISULFATE	7646-93-7	23
POTASSIUM SULFATE	7778-80-5	32
MAGNESIUM CARBONATE	546-93-0	2

(Continued)

HAZARDS IDENTIFICATION

Potential Health Effects

Causes skin, eye, nose, and throat irritation. May cause allergic skin reactions at high concentrations in sensitive individuals. Ingestion may cause inflammation and damage to the lining of the stomach, resulting in bleeding.

HUMAN HEALTH EFFECTS:

Skin contact may cause skin irritation with discomfort or rash. Allergic skin reactions were observed at high concentrations, but at lower concentrations of 12 ppm and 150 ppm, no allergic reactions were noted. Eye contact may cause eye irritation with discomfort, tearing, or blurring of vision. Inhalation may cause irritation of the upper respiratory passages with coughing and discomfort. Ingestion may cause gastritis possibly progressing to necrosis or hemorrhage.

Individuals with preexisting diseases of the skin or gastrointestinal tract may have increased susceptibility to the toxicity of excessive exposures.

Carcinogenicity Information

None of the components present in this material at concentrations equal to or greater than 0.1% are listed by IARC, NTP, OSHA or ACGIH as a carcinogen.

FIRST AID MEASURES

First Aid INHALATION

If inhaled, remove to fresh air. If not breathing, give artificial respiration. If breathing is difficult, give oxygen. Call a physician.

SKIN CONTACT

In case of contact, immediately wash skin with soap and water. Wash contaminated clothing before reuse.

EYE CONTACT

In case of contact, immediately flush eyes with plenty of water for at least 15 minutes. Call a physician.

INGESTION

If swallowed, do not induce vomiting. Immediately give 2 glasses of water. Never give anything by mouth to an unconscious person. Call a physician.

(Continued)

FIRE FIGHTING MEASURES

Flammable Properties
Will not burn.

Fire and Explosion Hazards:

Storage of large masses of "OXONE" can trap heat and lead to ignition of paper bags. Grinding or intensive mixing may cause ignition of oxidizable material present.

Extinguishing Media
Water.

Fire Fighting Instructions
None.

ACCIDENTAL RELEASE MEASURES

Safeguards (Personnel)
NOTE: Review FIRE FIGHTING MEASURES and HANDLING (PERSONNEL) sections before proceeding with clean-up. Use appropriate PERSONAL PROTECTIVE EQUIPMENT during clean-up.

Accidental Release Measures
Sweep up. Flush area with low pressure water.

HANDLING AND STORAGE

Handling (Personnel)
Avoid breathing dust. Avoid contact with eyes, skin, or clothing. Wash thoroughly after handling.

Storage
Store in a cool, dry, well-ventilated area. Stack on pallets providing air space; closely stacked bags should not exceed a 4 ft. (1.2 m) cube. Keep packages dry. Do not store with combustible materials.

EXPOSURE CONTROLS/PERSONAL PROTECTION

Engineering Controls
Use sufficient ventilation to keep employee exposure below recommended limits.

Personal Protective Equipment
EYE/FACE PROTECTION

Wear safety glasses. Wear coverall chemical splash goggles and face shield when the possibility exists for eye or face contact from airborne material.

RESPIRATORS

(Continued)

EXPOSURE CONTROLS/PERSONAL PROTECTION(Continued)

A NIOSH/MSHA approved air-purifying respirator with a dust/mist cartridge or canister may be permissible under certain circumstances where airborne concentrations are expected to exceed exposure limits. Protection provided by air purifying respirators is limited. Use a positive pressure air supplied respirator if there is any potential for an uncontrolled release, exposure levels are not known, or any other circumstances where air purifying respirators may not provide adequate protection.

PROTECTIVE CLOTHING

Wear impervious clothing, such as gloves, apron, boots or whole bodysuit, made of rubber, as appropriate. Leather gloves may be used when handling dry material.

Exposure Guidelines**Exposure Limits****"OXONE" MONOPERSULFATE COMPOUND**

PEL (OSHA)	Particulates (Not Otherwise Regulated) 15 mg/m ³ , 8 Hr. TWA, total dust 5 mg/m ³ , 8 Hr. TWA, respirable dust
TLV (ACGIH)	None Established
AEL * (Du Pont)	1 mg/m ³ , 8 Hr. TWA

Other Applicable Exposure Limits**POTASSIUM SULFATE**

PEL (OSHA)	None Established
TLV (ACGIH)	None Established
AEL * (Du Pont)	10 mg/m ³ , 8 Hr. TWA

MAGNESIUM CARBONATE

PEL (OSHA)	15 mg/m ³ , total dust, 8 Hr. TWA 5 mg/m ³ , respirable dust, 8 Hr. TWA
TLV (ACGIH)	10 mg/m ³ , total dust, 8 Hr. TWA
AEL * (Du Pont)	None Established

* AEL is Du Pont's Acceptable Exposure Limit. Where governmentally imposed occupational exposure limits which are lower than the AEL are in effect, such limits shall take precedence.

PHYSICAL AND CHEMICAL PROPERTIES**Physical Data**

Boiling Point	@ 760 mm Hg Decomposes
Vapor Pressure	Nil
Vapor Density	Not volatile
Melting Point	Decomposes
Evaporation Rate	(Butyl acetate = 1) Not volatile
Solubility in Water	25.6 WT% @ 20 C (68 F)
pH	1% solution = 2.3; 3% solution = 2.0
Odor	Odorless
Form	Granular; free flowing solid
Color	White
Specific Gravity	1.1-1.4

(Continued)

STABILITY AND REACTIVITY

Chemical Stability

Stable.

Incompatibility with Other Materials

The mixture of "OXONE" with compounds containing halides or active halogens can cause release of the respective halogen if moisture is present. For example, mixture with sodium dichloroisocyanuride or with sodium chloride can cause release of chlorine gas; mixture with cyanides can cause release of hydrogen cyanide gas; and heavy metal salts such as those of cobalt, nickel, copper, or manganese cause the evolution of oxygen.

Decomposition

Releases oxygen gas.

Polymerization

Polymerization will not occur.

TOXICOLOGICAL INFORMATION

Animal Data

Inhalation 4-hour LC50: >5 mg/l in rats
Skin absorption LD50 : >11,000 mg/kg in rabbits
Oral LD50 : 2,000 mg/kg in rats

The compound is a severe skin and eye irritant, but is not a skin sensitizer in laboratory animals. Single inhalation exposures produced nonspecific effects such as weight loss and irritation. Repeated inhalation exposures produced eye irritation and reversible corneal damage. By ingestion, the administration of large single doses produced nonspecific effects such as weight loss and irritation as well as gastric ulceration, necrosis, and hemorrhage. The compound does not produce genetic damage in bacterial cell cultures.

ECOLOGICAL INFORMATION

Ecotoxicological Information

Aquatic Toxicity

Potassium Sulfate

96-hour Tlm, bluegill sunfish : 3,500 mg/L

Magnesium Carbonate

96-hour LC50, species unidentified: >1,000 ppm

(Continued)

DISPOSAL CONSIDERATIONS

Waste Disposal

Comply with Federal, State, and local regulations. If approved, flush to sewer or waste treatment plant. Large quantities should be neutralized with soda ash.

TRANSPORTATION INFORMATION

Shipping Information

NOT REGULATED AS A HAZARDOUS MATERIAL BY DOT OR IMO.

Shipping Containers

Multiwall Bags
Fiber Pack Drums

REGULATORY INFORMATION

U.S. Federal Regulations

TSCA Inventory Status Reported/Included.

TITLE III HAZARD CLASSIFICATIONS SECTIONS 311, 312

Acute : Yes
Chronic : No
Fire : No
Reactivity : No
Pressure : No

LISTS:

SARA Extremely Hazardous Substance - No
CERCLA Hazardous Material - No
SARA Toxic Chemical - No

CANADIAN WHMIS CLASSIFICATION:

D2B

OTHER INFORMATION

NFPA, NPCA-HMIS

NPCA-HMIS Rating	
Health	2
Flammability	0
Reactivity	1

Personal Protection rating to be supplied by user depending on use conditions.

Additional Information

For further information, see DuPont "OXONE" Monopersulfate Compound Data Sheet.

(Continued)

The data in this Material Safety Data Sheet relates only to the specific material designated herein and does not relate to use in combination with any other material or in any process.

Responsibility for MSDS	DuPont Chemicals
Address	Engineering & Product Safety P. O. Box 80709, Chestnut Run Wilmington, DE 19880-0709
Telephone	302-999-4946

Indicates updated section.

End of MSDS

Test Log and Data Sheets

Appendix E

MODEL		TITLE	OROME DISSOLUTION RATE	BY	WADSWORTH
FILE			TABLET # 1	DATE	17 JUN 94
JOB				PAGE	OF

H₂O FLOW RATE 25 ML/SEC
 FLOW TIME 20 SEC
 H₂O QUANTITY/CYCLE 500 ML
 AIR FLOW RATE 10 CFM

CYCLE	TEMP OF	P _H	+ TARE WEIGHT (g) READING Δ	Air Weight
0			5.81	5.13
①			3.63	4.95
②	104.4		5.21	4.33
③	104.4		4.40	3.72
④	101.2		4.02	3.34
⑤	98.6		3.52	2.84
⑥	98.3		2.94	2.26
⑦	98.4	NOT	2.54	1.86
⑧	98.4	RECORDED	1.91	1.23
⑨	98.2		1.89	1.21
⑩	98.3		1.59	0.91
⑪	98.3		1.40	0.72
⑫	98.4		1.17	0.49
⑬	98.3		0.91	0.23
⑭	98.2		0.78	0.10

TARE WEIGHT 0.30 g

RESIDUE WEIGHT
 Residual P_H = 4

MODEL		TITLE	OXONE Dissolution RATE	BY	D.D. MATHIE
FILE		TEST	TABLET # 2	DATE	18 NOV 94
JOB				PAGE	OF

20 ml/sec x 20 SEC = 400 ml
10 CFM

START	WEIGHT OF	WETTED PH	Flow STOP	WEIGHT (g)	BAG + FILTER
				11.97	
①	92.2	~4/4		11.73	Δ 0.24 5.59
②	98.5	4		10.88	Δ 0.85 5.35
③	98.5	3 1/2		10.81	Δ 0.07 4.50
④	98.8	4		9.40	Δ 1.40 4.43
⑤	98.8	4		8.74	Δ 0.66 3.03
⑥	98.7	4		8.42	Δ 0.32 2.37
⑦	98.8	4		7.99	Δ 0.43 2.05
⑧	98.5	3 1/2		7.47	Δ 0.52 1.62
⑨	98.5	4 1/2		7.27	Δ 0.20 1.10
⑩	98.7	4 1/2		6.96	Δ 0.31 0.90
⑪	98.5	4 1/2		6.81	Δ 0.15 0.59
⑫	98.7	5		6.71	Δ 0.10 0.44
⑬	98.9	4 1/2		6.55	Δ 0.16 0.34
⑭	98.7	4 1/2		6.48	Δ 0.07 0.18
⑮					0.11

Residue weight 0.11 g

DRY WEIGHT OF
FILTER & BAG 6.00 g



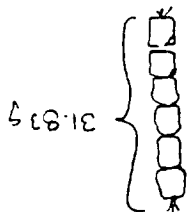
MODEL		TITLE	OXIDE EMISSION DATE TEST
FILE	WPI'S	DATE	23 JUN 94
JOB		PAGE	1 of 2

Pin type OXIDE / OET WFR 1 JUN 94

Pin weight

#1 5.31 g
#2 5.37 g
#3 4.57 g
#4 4.77 g
#5 5.98 g
#6 5.66 g

31.66 g



Pin weight

Sock = 5.58 g
Pic Assy = 31.83 g

37.41 g

38.08 g
WET
DAY

DP

NO FURN (with FURN)
0.8 INLET H₂O
1.7 INLET H₂O

FURN Assy DP = 0.9 IN H₂O
DAY

POST TEST DP 1.6 IN H₂O WET

POST TEST PH = 3

FORM H₂O THRU OUT = 9.33 LITERS

H₂O FLOW RATE (READING (%)) ACTUAL

52 %

20 ml/sec

H₂O FLOW TIME

20 SEC

Air FLOW RATE

READING

ACTUAL

0.5 IN H₂O

10 CFM

HAWAII CITY FLOW PER CYCLE
9.330 LITERS / 20 CYCLES = 466.5 ml/cycle



UNITED
TECHNOLOGIES
HAMILTON
STANDARD

MODEL		TITLE	PIN EMISSION TEST	BY	D. W. RYKLE
FILE	UPIS		IN DI WATER	DATE	23 NOV 94
JOB				PAGE	2 OF 2

CYCLE #	TEMP (°F)	PH	WEIGHT		Pin Weight
			Tot. Reading	Δ(g)	
0			38.08		31.66
①	97.8	2 1/2	36.08	Δ 2.05	29.66
②	98.3	2 1/2	33.43	Δ 2.65	27.01
③	98.2	2 1/2	30.24	Δ 3.19	23.82
④	98.2	2 1/2	27.53	Δ 2.71	21.11
⑤	98.4	2 1/2	24.94	Δ 2.59	18.52
⑥	98.3	2 1/2	22.40	Δ 2.54	15.98
⑦	98.3	3 - 4 *	20.01	Δ 2.39	13.59
⑧	98.4	3 1/2	17.82	Δ 2.19	11.40
⑨	98.2	3	15.81	Δ 2.01	9.39
⑩	98.2	3	14.38	Δ 1.43	7.96
⑪	98.2	3	12.79	Δ 1.59	6.37
⑫	98.2	3	11.72	Δ 1.07	5.30
⑬	98.2	3	10.74	Δ 0.93	4.37
⑭	98.2	3 1/2	9.43	Δ 0.86	3.51
⑮	98.2	3	8.98	Δ 0.95	2.56
⑯	98.3	3	8.56	Δ 0.42	2.14
⑰	98.8	4	8.07	Δ 0.49	1.65
⑱	98.3	4 1/2	7.69	Δ 0.28	1.37
⑲	98.4	4 1/2	7.40	Δ 0.29	1.08
⑳	98.4	4	7.19	Δ 0.21	0.87

Test Plan
For
Urine Pretreat
Injection System

Appendix E



TEST PLAN

FOR

URINE PRETREAT INJECTION
SYSTEM
(UPIS)

30 DAY PROTOTYPE
EVALUATION FOR OXONE®
INTRODUCTION

FEBRUARY 1995

Prepared by:

Approved by:

A handwritten signature in black ink, appearing to read "D.W. Rethke", written over a horizontal line.

Donald W. Rethke
Senior Principal Engineer

A horizontal line with a small flourish at the end, intended for a signature.

Test Title

Urine Pretreat Injection System (UPIS-1) 30-day prototype evaluation test for oxone® introduction.

- Test objective:** The purpose of this test program is to evaluate the prototype solid (tablet) injection method selected for introducing proper levels of oxone safely and conveniently into a micro-gravity two-phase urine/air intrainment collection system.
- Test Hardware:** Test equipment to be used is the Shuttle DTO two-phase urine fan/separator with the clear lexan housing installed. The test fan/separator will be installed in the portable urine collection test rig (known as the Wizz on Wheels), which will be located conveniently in a men's bathroom in Building 1A at Hamilton Standard.
Also used will be the DTO flight type dual check valve, which is located in the urine outlet line.
- Test Setup:** The portable urine collection test setup will be positioned in one of the bathrooms to collect urine from volunteers on a real-time basis. Fresh, warm urine will be processed immediately through the DTO urine fan/separator and pumped into a downstream pressurized accumulator. See Schematic in Figure 2. A prototype pretreat/prefilter will be installed in the urine collection inlet housing and will be changed on a routine time line. Provisions will be provided for downstream urine with dissolved oxone to be stored and analyzed. An electrical block diagram is also shown in Figure 4, which shows the basis electrical hookup.
- Test Procedure:** The detailed test procedure for the 30-day urine collection test is divided into several tasks, which include the following:
- Test Rig Checkout - The portable urine test rig will be initially checked out using water as a test media to verify that the mechanical and electrical elements of the rig operate properly. During this test the oxone pretreat-prefilter will not be used. The checkout will also be used as a basepoint test to determine if any degradation of the test hardware has occurred during the 30-day tests. (A basepoint test will be conducted after completion of the test before teardown). Data will be taken of various parameters of the urine fan/separator operation such as speed, output pressure, and, etc.
- Also, as part of rig checkout, a baseline of the dissolution rate of the prototype pretreat/prefilter will be conducted. The latest version of the prototype pretreat/prefilter will be installed in the inlet hose and water will be introduced into the system at the same rate, quantity, and cycle time as the previous laboratory dissolution rate test procedure. This test will be conducted to determine if there are any differences between the lab test setup and the portable urine collection test set.

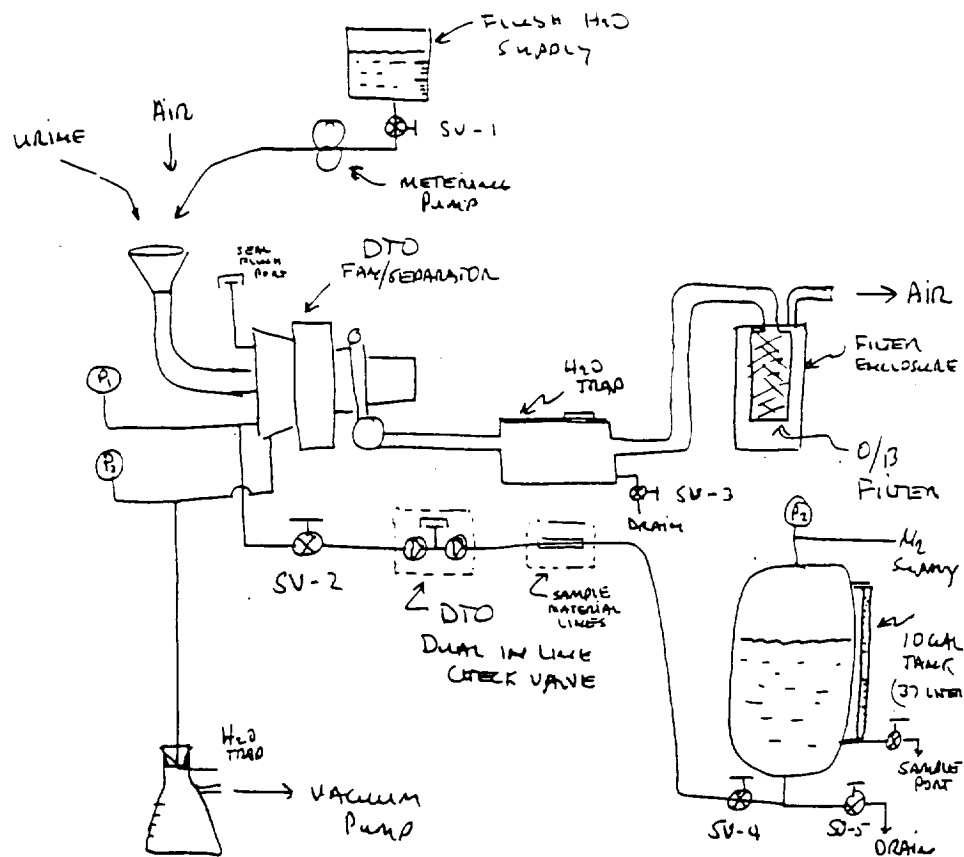


Figure 2 Schematic of Portable Urine Collection Test Rig

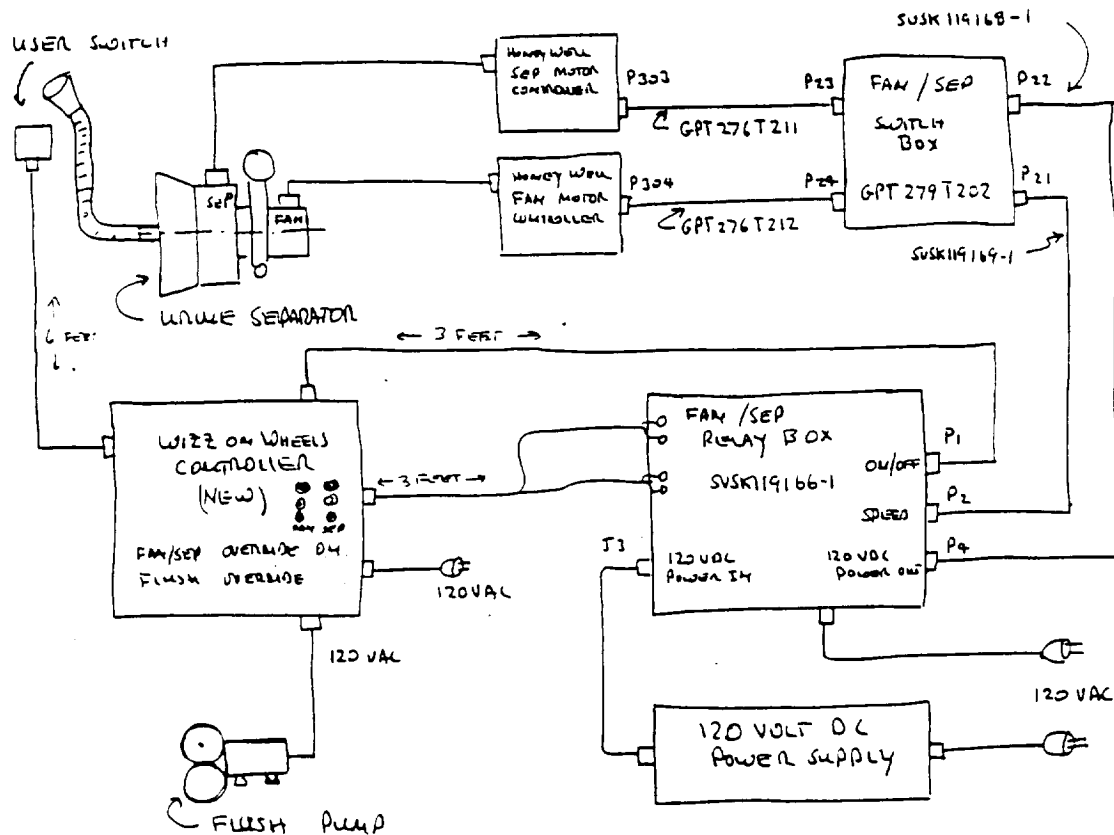


Figure 4 Electrical Block Diagram for Wizz on Wheels UPIS Test

User Instructions

1. Verify "ready to use" sign.	
2. Lift "anti-use" lid.	Note: This action will turn on the fan/separator.
3. Verify two (2) green lights.	<p>Note: This indicates that both fan and separator are at operating speed.</p> <p>If both green lights are not on:</p> <ul style="list-style-type: none">• Do not use test item• Close lid• Use normal urinal• Call Dr. Flush (x2166)
4. Do your thing!!	
5. Close lid when done.	Note: This action will automatically provide an 80ml H ₂ O flush and shut down the fan/separator after a one-minute fan dry-out cycle.

User Helpful Hints

- Please try to keep collection area clean.
- Please only one cycle of the lid per use. (Extra cycles will offset test calculations.)
- If a line forms -- next user is requested to wait until fan/separator shut down prior to relifting lid.
- If you goof, it will not be held against you, but please let Dr. Flush know so that the test data can be properly compensated for.
- Your comments are welcome!!

Thank you very much
for your participation
Dr. Flush (x2166)

Figure 5 User Instructions

UPIS 30 DAY EVALUATION TEST			
BATCH #			
PARAMETER	INITIAL	FINAL	Δ
TIME & DATE (— AM/PM —/—/—)			
S/N OF PRETREAT/PREFILTER INSTRUMENT			
STOP/START CYCLE READOUT (#)			
TEST RIG COUNTER (#)			
SEDIMENTATION RUNNING TIME (HOURS)			
ACCUMULATOR PRESSURE (PSIG)			
WEIGHT OF URINE COLLECTED (LBS)			
PH OF URINE COLLECTED (PH)			
200 ML SAMPLE I.D. NUMBER (#)			
NUMBER OF PILLS TOTALLY DISSOLVED		— OF —	
WEIGHT OF PRETREAT (GRAMS)			
VISUAL STATUS OF TRANSPARENT LINES			
OTHER NOTES & COMMENTS: _____			
FLUSH VOLUME USED (ML)	$(\text{CYCLES}) \times (80 \text{ ML/CYCLE}) = \boxed{} \text{ ML}$		
RATIO OF OXONE/URINE → FLUSH	$\frac{() \text{ g OXONE USED}}{() \text{ LITERS}} = \boxed{} \text{ g/L}$		
AVERAGE VOID (ML/CYCLE)	$\frac{(\text{ML TOTAL COLLECTED}) - (\text{ML FLUSH})}{(\text{CYCLES})} = \boxed{} \text{ ML}$		

Figure 6 Sample Data Sheet

Test completion and tear down

Upon completion of the 30-day urine collection test (or longer if time and need allows) a post test base point test similar to the pretest base point will be conducted using water. The pre and post test data will be compared to determine any significant changes. Also, the clear tygon tubes and the clear lexon housings on the fan/separator will be visually inspected for evidence of contamination or urine solids buildup. If required the fan/separator will be disassembled for further internal visual inspection along with photographs.

APPENDIX A

LAB PROCEDURES

Chemical Analysis of Pretreated Urine

The following analyses will be performed in the Chemical Analysis Laboratory.

Quantity: 200 mL

Container: Plastic Falcon Disposable Cup with Lid

Analyses:

pH: pH will be measured using an Orion epoxy-body electrode.

Conductivity: Conductivity will be measured using a YSI Conductivity Bridge.

Active Oxygen: Active oxygen content (or Oxone content) will be determined by titrating with sodium thiosulfate. This is the analysis procedure recommended by DuPont. The analysis procedure is outlined below.

Titration: Iodine released from an acidic potassium iodide solution by the active oxygen in the Oxone is with standard sodium thiosulfate to a colorless endpoint.

1. Use enough pretreated urine to contain approximately 0.5 g of dissolved oxone. Weight to the nearest 1 mg.
2. In a 250 mL Erlenmeyer flask, combine the urine sample, 5 mL of 20% sulfuric acid and 10 mL of 20% potassium iodide. Add two drops of starch solution. Mix well.
3. Titrate with 0.1 N sodium thiosulfate to a colorless endpoint that persists for 30 seconds.
4. Calculation:

$$\% \text{ Active Oxygen} = \frac{Q \cdot N \cdot 0.008 \cdot 100}{M}$$

Where: Q = Volume of sodium thiosulfate (mL)
N = Exact normality of sodium thiosulfate
M = Mass of sample (g)

$$\% \text{ Active component (KHSO}_5\text{)} = \frac{\% \text{ Active Oxygen}}{0.1053}$$

Oxone Concentration Evaluation
In Urine

Appendix G

Internal Correspondence



March 28, 1995
SVME:3445

Memorandum to: D. Rethke

cc: J. Steele, J. Varsik, B. Peyton

From: R. Marsh

Subject: Oxone Concentration Evaluation In Urine

.....

The purpose of this memorandum is to report the laboratory findings for the evaluation of Oxone® as a urine additive to control microbial growth.

Introduction

Oxone® is a monoperoxysulfate compound that has many applications where a strong oxidant is required in cleaning and bleaching uses. The Oxone® data sheet is attached. This material is being evaluated as a pretreatment in the International Space Station Alpha (ISSA) Waste Collection System (WCS) for crew urine. Chemical treatment of urine will be required on ISSA to prevent the development of undesirable noxious odors, proliferation of microorganisms, and the prevention of urine salt formation. This study evaluated the effect of several concentrations of Oxone® to control microbial growth in a pooled male human urine.

The experiment challenged the urine with Pseudomonas aeruginosa and Pseudomonas cepacia test organisms. This was done to simulate a worst case scenario which could simulate a crew member with a urinary tract infection or any other event where microbes could be introduced to the WCS.

Oxone® was added to the urine at concentrations of 0.1, 1.0, 2.0, 3.0, 4.0, and 5.0 grams per liter (gm/L). The target use concentration for Oxone® in the WCS is 5.0 gm/L.

The interest that initiated this study was how well will Oxone® control microbial growth and will the 5.0 gm/L be adequate.

Results

The urine containing Oxone® and the challenge microorganisms was maintained at ambient temperature and was assayed at time 0 and at 3 and 7 days. The urine containing Oxone® 0.1, 1.0 and 2.0 gm/L showed no effect controlling the microbial challenge and the population increased. At 3.0 gm/L Oxone® there was limited control which allowed further microbial development. The 4.0 and 5.0 gm/L Oxone® levels prevented microbial growth throughout the test period. This data is illustrated in the attached graph and data sheet. The attached photographs illustrate the various test concentrations and show the

effect of microbial control vs. no control. Photograph 1 shows the range of the Oxone® concentrations (0 to 5 gm/L), effective microbial control can be seen in bottles labeled 4.0 and 5.0. The bottle's label on the reverse side of the tape label is clearly visible through the treated urine (no microbial growth). The urine in the other bottles (0 to 3.0) is very turbid indicating no microbial control and the labels can not be seen, bottle 3.0 shows partial microbial control. Photograph 2 shows these observations in greater detail.

The pH of these samples was measured on test day 11. The pH values ranged from 8.94 for 0.1 gm/L Oxone® to 4.48 for 5.0 gm/L.

Discussion

This test evaluated Oxone® as a microbial control agent at six concentration levels. Microbial control was attained at 4.0 and 5.0 gm/L in the pooled male human urine. The Oxone® concentrations less than 4.0 gm/L were not effective controlling microbes and high populations developed. The urine collected on ISSA is planned to be processed on a daily basis. Therefore, the Oxone® treatment at greater than 4.0 gm/L appears to be adequate to control microbes in the WCS.

This study was not able to observe urine salt formation since the urine was collected and held for 18 hours at 4°C after collection and prior to use the urine was filtered through glass wool to remove any precipitates. The test was run at ambient temperature which would not be favorable for salt precipitation. No urine salts were visible in the test bottles during the test period.

Recommendation

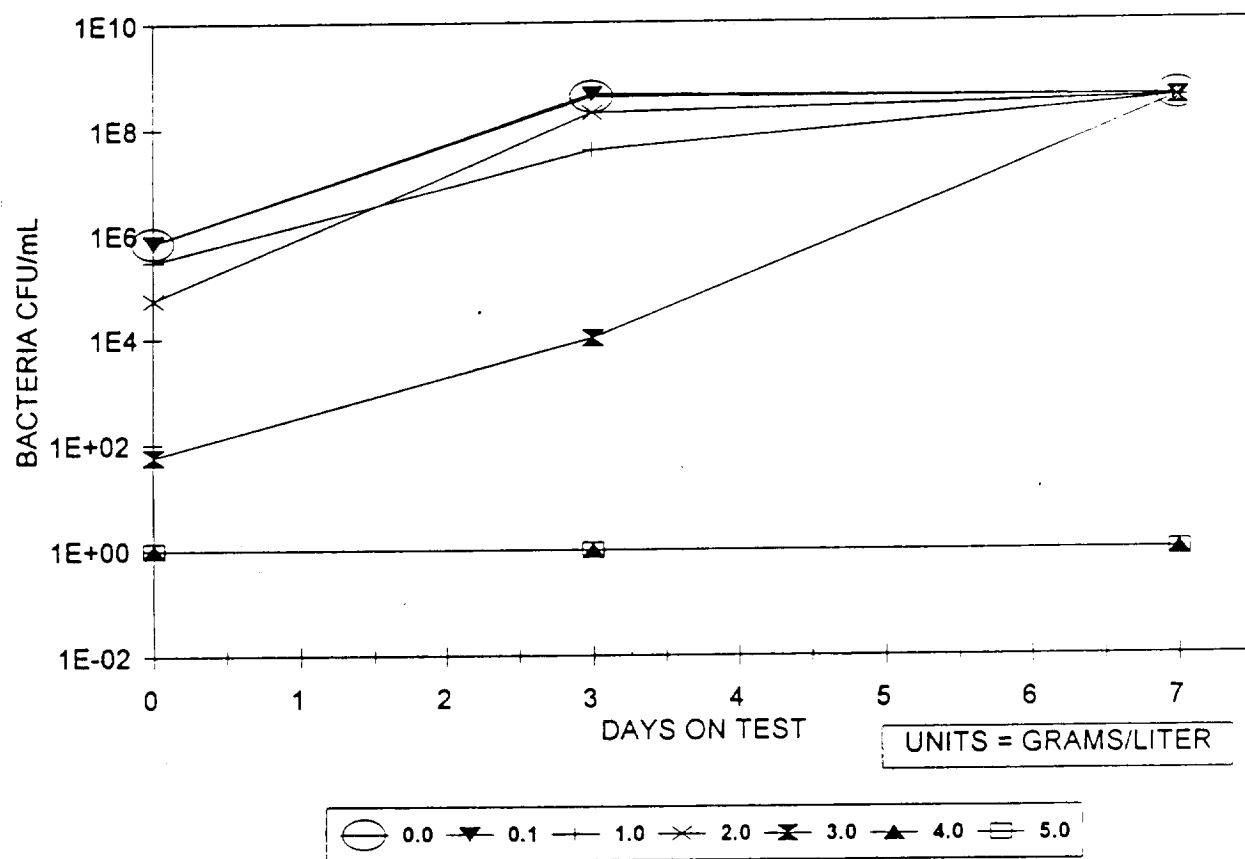
Based upon this study Oxone® was shown to be effective at the 4.0 to 5.0 gm/L concentration in controlling microbes in human urine. This concentration should be maintained throughout the WCS to prevent the development of microorganisms that could pose a problem by generating odors or increasing the microbe level within the cabin.

Prepared by: *Robert W. Marsh*
Robert W. Marsh

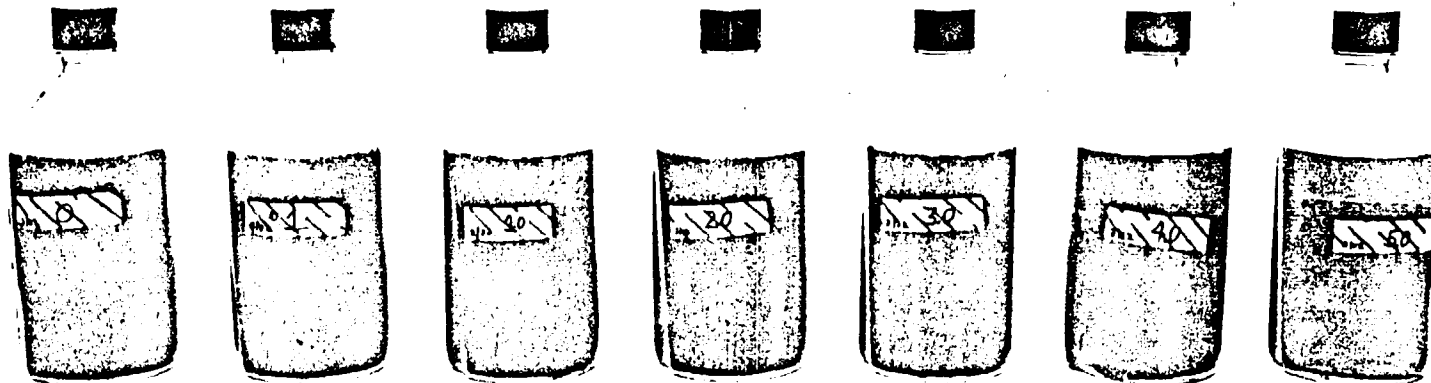
Attachments:

- Oxone® Data Sheet
- Graph
- Data Sheet
- Photographs

URINE PRETREATMENT INJECTION SYSTEM OXONE AS A MICROBIAL CONTROL AGENT



G-5



DATE	DAYS ON TEST	OXONE 0	OXONE 0.1	OXONE 1	OXONE 2	OXONE 3	OXONE 4	OXONE 5	BLANK 0	GRAMS/LITER
02/24/95	0	6.80E+05	7.00E+05	3.00E+05	5.50E+04	6.00E+01	1.00E+00	< 1.00E+00	4.80E+04	
02/27/95	3	4.00E+08	4.40E+08	3.90E+07	2.00E+08	1.00E+04	< 1.00E+00	< 1.00E+00		
03/03/95	7	3.80E+08	3.70E+08	3.50E+08	3.50E+08	3.50E+08	< 1.00E+00	< 1.00E+00		
pH VALUE										
03/07/95	11	8.96	8.94	6.88	6.7	5.36	4.93	4.48		

URINE PRETREAT INJECTION SYSTEM
FEBRUARY 1995
OXONE CONCENTRATION EVALUATION

**Urine Pretreatment Injection
System - Microbial Air Sampling**

Appendix H

Internal Correspondence



May 4, 1995
SVME: 3477

Memorandum to: D. Rethke

cc: J. Steele, J. Varsik,

From: R. Marsh

Subject: Urine Pretreatment Injection System - Microbial Air Sampling

.....

The purpose of this memorandum is to report the laboratory findings for the air sampling that was done on the Urine Waste Collection System.

Introduction

There is a valid interest and concern about the microbial air quality within closed environments. Space vehicles in particular are of a great concern due to their re-circulated air, the crew is confined within the cabin for the mission duration, and due to the vehicle's remote location it is without immediate help or relief. Within a space vehicle, the life support systems could be microbe generators due to microbe growth on biological waste materials. This study examined the microbial content of air at points entering and within the Urine Waste Collection System. The Urine Waste Collection System studied used Oxone® as a urine pretreatment to control microbial activity in urine within the system. The effectiveness of Oxone® as a microbial control agent was described in SVME 3445.

The study sampled air with a Millipore® All-Glass Impinger at an air flow rate of 9.2 liters per minute for 10 minute periods. The microbes were collected in an impingement fluid described by Millipore® that was assayed for microbial content using R2A media. The air sampling points were: 1 - at the funnel entry of the system, 2 - between the urine separator and the filter, and 3 - at the air outlet after the filter.

Results

The results of the study showed that the sample points within the system and at the system air outlet did not contain microbes over the detection limits of 12 CFU per M³. The air sampled at the funnel entry point showed that the room air entering the system contained 193 CFU per M³. The recovered microorganisms were of several different types.

<u>Sample Point</u>	<u>Result</u>
System Inlet	193 CFU/M ³
Between Separator & Filter	
After urine collection	<12 CFU/M ³
After water flush	<12 CFU/M ³
System Outlet	<12 CFU/M ³

Conclusion

These results indicate that the Urine Waste Collection System using Oxone® as a urine pretreatment for microbial control does not add microbes to the effluent air over the environmental back ground within the time frame of this test. The Oxone® pretreatment was demonstrated to be an effective microbial control as described in SVME 3445. In this testing, there were no microbes recovered over the detection limits from the air within or exiting the Urine WCS system.

Prepared by: 
Robert W. Marsh

